2015–1950, 2015-1967

UNITED STATES COURT OF APPEALS FOR THE FEDERAL CIRCUIT

U.S. WATER SERVICES, INC. AND ROY JOHNSON,
Plaintiffs–Appellants,

v.

NOVOZYMES A/S AND NOVOZYMES NORTH AMERICA, INC.,
Defendants–Cross-Appellants.

Appeal from the United States District Court for the Western District of Wisconsin in case no. 3:13-cv-00864-jdp, Judge James D. Peterson

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November 2, 2015

CERTIFICATE OF INTEREST

Counsel for appellants U.S. Water Services, Inc. and Roy Johnson certify the following:

The full name of every party represented by me is:

U.S. Water Services, Inc. and Roy Johnson

The names of the real parties in interest represented by me are:

U.S. Water Services, Inc. and Roy Johnson

The names of all parent corporations and any publicly held companies that own 10% or more of the stock of U.S. Water Services, Inc. represented by me are:

Allete, Inc.

The names of all law firms and the partners or associates that appeared for the party or amicus curiae now represented by me in the trial court or are expected to appear in this Court are listed below.

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Novozymes	Defendants-cross-appellants Novozymes A/S and Novozymes North American, Inc.
Order	The district court's July 29, 2015 summary judgment order, ECF No. 561 (A2-37).
S.J. Opp'n	Plaintiffs' Opposition to Novozymes' Motion for Summary Judgment, ECF No. 226 (A1636-1823)
Veit	World Intellectual Property Application No. WO 2001/62947 to Chris Veit et al. (A1581-1610)
U.S. Water	Plaintiffs-appellants U.S. Water Services, Inc. and Roy Johnson
'137 patent	United States Patent No. 8,415,137 (A134-42)
'399 patent	United States Patent No. 8,609,399 (A143-52)
'### (xx:yy-zz)	Column xx, lines yy to zz of the '### patent

STATEMENT OF RELATED CASES

U.S. Water and its counsel are unaware of any other appeals in or from the same civil action that have previously been before this or any appellate court. U.S. Water and its counsel are likewise unaware of this or any other cases pending in this or any other court that will directly affect or be directly affected by a decision in this case.

INTRODUCTION

As the district court noted, correctly, "[t]he issue on which this case ultimately turns is whether [prior art references] Veit and Antrim inherently disclose using phytase to reduce deposits in ethanol production machinery." A26 (Order at 25). The court further noted, again correctly, that "[w]hether a limitation is inherent in the prior art is a question of fact." A26 (Order at 25) (citing *SmithKline Beecham Corp. v. Apotex Corp.*, 403 F.3d 1331, 1343 (Fed. Cir. 2005)). Thus, only "if the non-moving party fails to adduce evidence to genuinely dispute that a reference inherently discloses a limitation," is inherent anticipation "appropriately decided on summary judgment." A26 (Order at 25).

Federal Circuit case law is clear: if the prior art does not necessarily and inevitably result in the claimed deposit reduction, then the claims are not inherently anticipated as a matter of law. Here, as the district court found, "U.S. Water has adduced evidence … that practicing Veit and Antrim will not *always* result in deposit reduction." A28 (Order at 27). This evidence should have been enough to defeat summary judgment. Instead, based on a misunderstanding of both the asserted patents and this Court's inherent anticipation precedent, the district court granted summary judgment. It is this single reversible error that, when corrected, will set this case back on its proper course.

The district court characterized U.S. Water's argument against inherent anticipation as reliant on the fact that the prior art references were directed to a goal different than deposit reduction. A28 (Order at 27 ("According to U.S. Water, this 'particular goal' saves the patents-in-suit from being anticipated by the prior art.")). But U.S. Water's position is not—and has never been—that the prior art methods merely had a different *goal* than deposit reduction. U.S. Water's position is that, although both the prior art and U.S. Water's claimed methods involve the addition of phytase to the fuel ethanol process (albeit, for different reasons), the prior art methods do not inherently anticipate because they do not inevitably and invariably result in the deposit reduction required by the claims of U.S. Water's patents.

Furthermore, the district court misapplied precedent involving prior art with disclosed ranges that overlapped with those of the claimed invention. As fully discussed below, regardless of whether there are overlapping ranges, this Court has never strayed from the fundamental principle that inherency requires a showing of inevitability and invariability.

The bottom line is that in this case the prior art does not necessarily result in deposit reduction. And deposit reduction is required by the claims of the U.S. Water patents. Thus, there can be no anticipation by inherency unless the prior art always produces deposit reduction. But the district court recognized that phytase can be employed according to the prior art methods without achieving the claimed reduction in deposits. For the district court to grant summary judgment of anticipation in the face of this evidence was reversible error.

STATEMENT OF JURISDICTION

This is an appeal from a July 29, 2015 grant of summary judgment of invalidity by the district court, along with the underlying claim construction, and dismissal of U.S. Water's infringement claims as moot. The district court had jurisdiction pursuant to 35 U.S.C. §§ 1331 and 1338(a).

The district court entered judgment on July 31, 2015, and entered an amended final judgment on August 20, 2015. U.S. Water timely filed a notice of appeal of the amended final judgment on August 24, 2015. This Court has jurisdiction pursuant to 28 U.S.C. § 1295(a)(1).

STATEMENT OF THE ISSUE

Whether it is proper to find inherent anticipation on summary judgment when there are acknowledged factual disputes as to whether the prior art inevitably and invariably leads to the claimed invention.

STATEMENT OF THE CASE

When ethanol is produced commercially, severe and tenacious deposits form on the ethanol processing equipment, especially downstream of the fermentation tanks. A145 ('399 patent (1:34-43)), A959-61. Contrary to prevailing industry wisdom, the inventors of the patents-in-suit discovered that these deposits were largely made up of phytic acid and phytic acid salts (referred to herein as "phytic acid"). A296-301 (Johnson Dep. at 108:25-127:14), A371, A375-77, A413 (Young Dep. at 24:1-14, 41:22-46:9, 190:5-91:8); *see also* A1824-25. The '399 and '137 patents claim methods of using an enzyme called phytase to reduce the formation of these phytic acid deposits. A141-42 ('137 patent (claims)), A150-52 ('399 patent (claims)). Specifically, the patents claim the use of phytase for the reduction of phytic acid deposits under specified conditions, including, for example, pH conditions, temperature, and dosage. A141-42 ('137 patent (claims)), A150-52 ('399 patent (claims)).

U.S. Water markets a phytase product called pHytOUT®, which it sells to ethanol processing plants with instructions regarding how to use it to reduce phytic acid deposits. A1411-23, A1825. After learning about the successes of U.S. Water's pHytOUT®, Novozymes introduced a competing phytase product, Phytaflow®, which it also markets for the reduction of phytic acid deposits. A1041-57, A1355-56.

Prior to the patents-in-suit, phytase had been considered as a means of improving the efficiency of certain aspects of ethanol processing, namely liquefaction, saccharification, and fermentation. *See* A1007, A1021-22, A1580-1610 (Veit), A1582 (*id.* (1:4-9)), A1611-23 (Antrim), A1615 (*id.* (4:9-13, 4:35-37)). But, as described in more detail below, the prior art did not suggest the use of phytase for deposit control and, in fact, prior to the instant invention, phytase was not used to reduce deposits. A1007, A1021-22, A1044-45. And, more importantly, and as also described in detail below, the prior art use of phytase in ethanol plants likely had no effect on phytic acid deposits at all, much less *necessarily and inevitably* resulted in deposit reduction. A850-66, A1009, A1011-14, A1023-27.

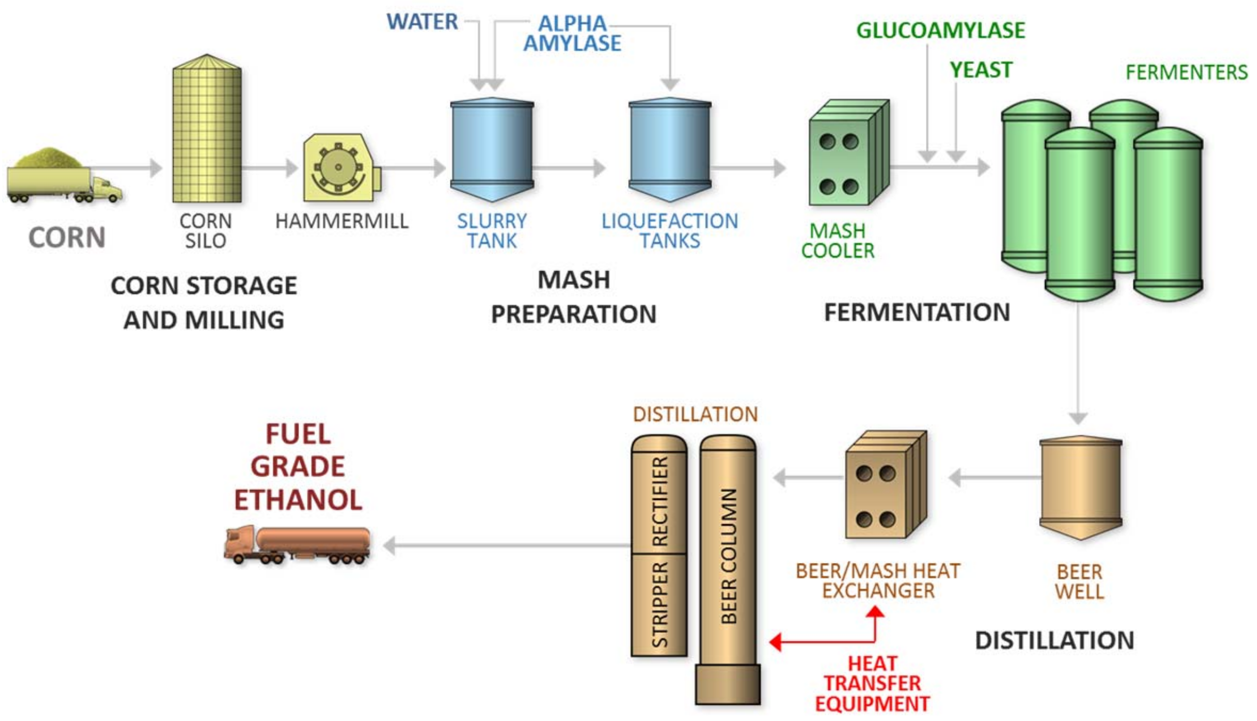
I. Statement of Facts

A. The ethanol production process.

Ethanol (like beer) is produced by using yeast to ferment starchy grains, such as corn or wheat. A771, A1250. In most United States ethanol plants, grains are milled to produce a meal, which is then mixed with water to form a mash.

A771-72, A1251-52. Enzymes (usually alpha amylases) are then added to the mash to begin the breakdown of starch into sugars. A771-72, A1251-52. The mash is then sent to tanks, called "liquefaction" tanks, to provide additional residence time for the enzymatic reactions. A771-72, A1252. After liquefaction, the slurry mash is cooled, and a second enzyme (glucoamylase) is added to complete the breakdown of starch into sugars. A771-72, A1253. This step is called "saccharification" and can be carried out while the mash is filling the fermentation tanks and continues throughout fermentation. A772, A1253. During fermentation, yeast is added, and the yeast metabolizes the sugars and produces ethanol. A772, A1253.

When fermentation is complete, the resulting product, referred to as "beer," is transferred into a holding tank (called a "beer well"), which holds the beer before it is sent to a distillation column, known as the "beer column." A772-73, A1254. In the beer column, ethanol is distilled from the beer. A773, A1254-55. Prior to entering the beer column, the beer must be heated to a very high temperature. A773, A1254. Thus, between the beer well and the beer column, the beer flows through a "beer-mash heat exchanger," where it is heated to a temperature from 130^0F to 150^0F. A773, A1254. In the beer column, the beer is further heated and the ethanol is distilled in the form of an ethanol-containing vapor. A773, A1254-55. An overview of the entire process is depicted below:


WATER
ALPHA AMYLASE
GLUCOAMYLASE
YEAST
FERMENTERS
CORN
CORN SILO
HAMMERMILL
SLURRY TANK
LIQUEFACTION TANKS
MASH COOLER
CORN STORAGE AND MILLING
MASH PREPARATION
FERMENTATION
FUEL GRADE ETHANOL
DISTILLATION
RECTIFIER
STRIPPER
BEER COLUMN
BEER/MASH HEAT EXCHANGER
BEER WELL
HEAT TRANSFER EQUIPMENT
DISTILLATION

B. Deposit formation (or fouling) is a common problem in ethanol production.

During ethanol production, deposits can form on ethanol processing equipment downstream of fermentation. A773-74, A1256-57. Such deposits generally form in heat transfer equipment, i.e., equipment where heat is transferred from one medium to another, such as the beer-mash heat exchanger and beer column. A773-74, A778-80, A1256-57. [redacted]

[redacted] In the industry, this phenomenon is commonly referred to as "fouling." A774-82, A1256-57, A1376-90.

Fouling impedes heat transfer and flow and compromises and renders less efficient the operation of mechanical devices used in ethanol processing. A155, A235, A778-80. It is a large and costly problem for the ethanol processing industry. A1378-80. Plants traditionally combated fouling using two methods. First, plants would clean the fouled equipment with acid or caustic; this was time consuming and expensive. A773-74, A1380, A1382, A480 (Fowler Dep. at 22:25-23:14). Second, at great cost, plants would increase the solubility of (and thus reduce deposit of) the deposit-forming material in the beer by adding large amounts of sulfuric acid to lower the pH of the beer. A773-74, A1380, A1382, A480 (Fowler Dep. at 22:25-23:14). [redacted]

[redacted] A1390.

C. Paul Young and Roy Johnson discovered a new way to reduce deposits.

Phytase is an enzyme that breaks down a molecule called phytic acid. A150 ('399 patent (12:30-57)), A752. In 2006 and 2007, U.S. Water, in particular Dr. Paul Young and Mr. Roy Johnson, began experimenting with use of phytase in ethanol processing. A296-98 (Johnson Dep. at 108:25-115:9), A371, A375-77 (Young Dep. at 24:1-14, 41:22-46:9). Dr. Young and Mr. Johnson had surprisingly discovered that the deposits believed for years to be magnesium *phosphate* actually contained magnesium *phytate*, which is a salt of phytic acid. A296, A300-01 (Johnson Dep. at 109:7-22, 125:6-126:14), A376-77, A413 (Young Dep. at 42:2-43:23; 190:5-191:8), A139-40 ('137 patent (Examples 1-2)). Indeed, for years, ethanol processing plant managers and designers had been attempting to resolve fouling under the incorrect belief that the source of fouling was magnesium phosphate. *See, e.g.*, A717 (Ferman Dep. at 42:7-14), A603-04 (Combs Dep. at 92:7-93:9), A650 (Gerry Dep. at 38:9-15).

Following this discovery, the inventors ran a further series of experiments to determine the impact of dosage, pH, time, and temperature on the use of phytase on these phytic acid deposits. A299-300 (Johnson Dep. at 120:24-123:24), A413-17 (Young Dep. at 190:5-209:7); *see also* A1344-53 ('244 patent (specification) (*see especially* Tbls. 1-4 and Examples 3-4)). Through their experiments, they realized that adding phytase to the processing fluid in a fuel ethanol plant could, depending on conditions such as concentration, temperature, residence time or reaction time, and pH, reduce the formation of phytic acid deposits in the plant. A146-50 ('399 patent (6:32-38, 11:65-67, Tbls. 1-4, and Examples 3-4)).

As with many important innovations, a significant part of the inventive process here was the inventors' discovery of the actual cause of the fouling problem that had been plaguing the fuel ethanol industry for so many years. The other part, equally important, was the development of a sophisticated recipe of phytase amounts, conditions, concentrations, and parameters of use in order to effectuate reduction in the formation of deposits.

D. The patents-in-suit pertain to new methods of using phytase for deposit control.

Mr. Johnson and Dr. Young filed their first patent application covering their invention on October 17, 2007. A301 (Johnson Dep. at 126:19-127:14), A1345 ('244 patent (face page)). The application described specific uses of phytase to reduce deposits of phytic acid in ethanol plants. *See generally* A1344-53 ('244 patent); *see also* A881-88. The specification of the patents-in-suit teaches that there are a number of conditions that affect the use of phytase to reduce phytic acid deposits:

> In an embodiment, the present invention provides a method in which the agent or enzyme is introduced into the ethanol-processing fluid under optimal conditions of temperature and pressure. Where the agent is phytase, the term "optimal conditions" refers to those conditions of concentration, temperature, residence time or reaction time, and pH that allow sufficient reaction with soluble phytate, phytate suspension, phytate precipitate, or insoluble phytate scale that reduces the level of the phytase deposit to an amount acceptable for operation of the ethanol plant or process. In an embodiment, the conditions provide for complete hydrolysis of soluble phytate and phytate suspension.

A138 ('137 patent (6:29-40) (highlighting added); *see also* A138 ('137 patent (6:10-13) ("phytase is added to ethanol processing equipment and/or processing fluid at a time point and under conditions required for the particular type of equipment or stage of ethanol processing")), A139, A140-41 ('137 patent (Tbls. 3 and 7) (showing that different concentrations of phytase release orthophosphate at significantly different rates)), A137-38 ('137 patent (Tbls. 1 and 2) (showing that the solubility of magnesium phytate varies with pH, temperature and the presence of ethanol)).

The United States Patent and Trademark Office ("PTO") issued to U.S. Water three patents claiming methods of reducing the formation of phytic acid and deposits on fuel ethanol processing equipment by the addition of a phytase enzyme. *See* A767, A1073-74, A1082-83, A1345, A1352-53 ('244 patent). The same PTO examiner examined each of the three patents. A1345 ('244 patent (face page)), A135 ('137 patent (face page)), A144 ('399 patent (face page)). The art she reviewed during her examinations included art related to prior use of phytase and included, for example, the same Veit and Antrim references relied on by the district court, discussed below. A1345 ('244 patent (face page)), A135 ('137 patent (face page)), A144 ('399 patent (face page)); *see also infra*, Statement of the Case, Part I.E.[1]

Two of these three patents have been asserted in this litigation, the '137 patent and the '399 patent. A153-81.[2] The '137 and '399 patents were issued to Roy Johnson and Paul Young on April 9 and December 17, 2013, respectively. A135 ('137 patent (face page)), A144 ('399 patent (face page)).[3]

All asserted claims of the '137 and '399 patents pertain to methods of adding phytase to ethanol processing fluid to reduce deposit formation of phytic acid. A141-42 ('137 patent (claims 1, 6, 12, 13)), A150-52 ('399 patent (claims 1, 2, 5-

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[1] United States Patent Publication Nos. 2002/0006647 and 2002/0187528 and United States Patent No. 7,244,597, all to Veit, were before the examiner in the prosecution of both patents-in-suit. A135 ('137 patent (face page)), A144 ('399 patent (face page)). At least 2002/0006647 claims priority to the same application as Application WO 2001/62947. *Compare* A1625 (2002/0006647 (face page) (claiming priority to PA 2000 00281)), *with* A1581 (Veit (face page) (also claiming priority to PA 2000 00281)).

[2] The '244 patent, which was the first of the three patents, issued on October 18, 2011, is not asserted in this action. A1345 ('244 patent (face page)), A153-81.

[3] U.S. Water has asserted claims 1, 6, 12, and 13 of the '137 patent and claims 1, 2, 5-12, 16-22, 25, 28-32, 34, and 35 of the '399 patent. A1829-32, A1838-39.

12, 16-22, 25, 28-32, 34, and 35)).[4] Importantly, each of the claims requires a reduction in the formation of phytic acid deposits. *See* A141 ('137 patent (claim 1) (including limitation "thereby reducing the formation of deposits of insoluble phytic acid or phytic acid salts on surfaces in the equipment")); A150 ('399 patent (claim 1) (including limitation "wherein [providing phytase] causes a reduction of the formation of insoluble deposits of phytic acid and/or salts of phytic acid in a piece of heat transfer equipment in the plant")).

E. Prior uses of phytase in ethanol production: Veit and Antrim.

As noted above, prior to the patents-in-suit, the use of phytase in ethanol production had been disclosed, but not for reducing the formation of deposits. A1007, A1021-22, A1044-45, A1582 (Veit (1:4-9)), A1615 (Antrim (4:9-13, 4:35-37)). Rather, phytase was described as potentially useful for *improving the*

[4] The different claims vary in regard to:

- the location where deposit reduction occurs (i.e., a piece of heat transfer equipment generally and/or specifically in the beer column);
- whether the pH of the ethanol processing fluid in the beer column during production of ethanol is 4.5 or higher;
- whether the reduction of phytic acid deposits is accomplished substantially without the addition of an acidic compound in the presence of an oxidizer, oxidizing agent, or ultraviolet light; and/or
- the concentration and activity of the phytase in the ethanol processing fluid.

A141-42 ('137 patent (claims 1, 6, 12, 13)), A150-52 ('399 patent (claims 1, 2, 5-12, 16-22, 25, 28-32, 34, and 35)).

efficiency of three other aspects in fuel ethanol plants: (1) liquefaction; (2) saccharification; and (3) fermentation. *See* A1007, A1021-22, A1582 (Veit (1:4-9)), A1615 (Antrim (4:9-13, 4:42-44)). The two prior art references at issue in this appeal, Veit and Antrim, teach using phytase for these purposes. A1007, A1021-22, A1582 (Veit (1:4-9)), A1615 (Antrim (4:9-13, 4:35-37)). They say nothing about the use of phytase to reduce the formation of deposits. A1007, A1021-22; *see generally* A1611-23 (Antrim), A1580-1610 (Veit). And, as noted above, both references were before the examiner in the prosecution of the patents-in-suit. A135 ('137 patent (face page)), A144 ('399 patent (face page)); *see supra* n.1.

1. Deposit reduction will only "switch on" *if* the appropriate phytase amount and process conditions are used.

To fully appreciate why Veit and Antrim do not disclose the conditions that will inevitably and invariably lead to reducing the formation of phytic acid deposits, it is first helpful to understand the kinetics of how such deposits form on heat transfer equipment in fuel ethanol plants. The kinetics of phytic acid fouling are critical when evaluating whether a plant would *necessarily* obtain fouling reduction simply because it added phytase somewhere in the ethanol production process.

The rate of phytic acid deposition on heat transfer surfaces is based on "zero order kinetics." A853. What this means is that under normal operating conditions, a plant will experience fouling at a constant rate *unless* a plant adds the appropriate type and amount of phytase, under the appropriate process conditions—such as taught by the patents-in-suit. A853-54. If the plant uses the right combination of phytase and process conditions, the plant will see fouling reduction "switch on" as the treated beer flows through the system. A853-54. However, if the right type and amount of phytase is not used and process conditions are not correct, fouling reduction *will not occur*. A854. This chemical trigger is particularly sensitive, meaning that slight variations in the phytase addition process can defeat its ability to reduce phytic acid at all. A854; *see also* A855-60, A862-63.

The chemistry of ethanol production does not allow a linear relationship between phytic acid concentration and fouling (deposits). A854-55. Thus, for example, removing half of the phytic acid in a plant does *not* cut the fouling rate by half. A854-55. That is, *one can remove a large majority of the phytic acid in a plant and still not have any measureable effect on the rate of process fouling*. A854. The following illustrates this phenomenon.

A typical ethanol plant has a range of twenty-four to thirty-six tons of phytic acid flow through it every single day. A850-51. Of this, only 1% or 2% of the phytic acid actually deposits on heat transfer equipment in the plant. A851-52. Thus, to begin reducing these deposits, the phytase must convert at least 98% of all the phytic acid flowing through the plant. A858-59. This means that even if a plant were able to eliminate most of the phytic acid from the ethanol solution, it would still be fully saturated and continue to form deposits. A859-60.

Whether phytase is able to break down substantially all of the twenty or thirty tons or more of phytic acid that flows through the system daily to achieve deposit reduction depends on whether the correct "recipe" is used. *See* A850-60. This means that, for a particular choice of phytase (different phytases all have different properties), the choice of the amount of phytase, the choice of the addition point, the choice of the residence time, and the choice of parameters such as pH and temperature must be coordinated such that the phytase can accomplish the critical task of converting enough phytic acid to effect a reduction in the formation of deposits. A750-62, A853-60. What's more, *each* of these choices can affect whether any given phytase will actually achieve the claimed reduction in the formation of deposits. A853-60, A760-62, A1008-14, A1023-26. In other words, one could add enough phytase to accomplish the purpose of Veit or Antrim and *never* see a reduction in the formation of deposits because there would still be too much phytic acid left in solution. A850-60, A1011-14, A1023-26.

2. The Veit reference.

PCT Application WO 2001/62947 to Veit ("Veit") entitled "Fermentation with a Phytase" was published on August 30, 2001. A1581-1610 (Veit). Veit is directed to improved saccharification and fermentation. A1007, A1582 (Veit (1:4-

9)). In particular, Veit describes the addition of phytase *for improving amylase action* during saccharification by removing the phytic acid-mediated inhibition of amylase enzymes, and improving fermentation efficiency by making more minerals available to the yeast cells. A1007-08, A1582 (Veit (1:4-9)), A1594-97 (Veit (13:1-16:7)).

a. Veit does not teach or result in deposit reduction.

Veit is not directed to the use of phytase to reduce the formation of deposits of phytic acid. A1007, A1582 (Veit (1:4-9)). Veit does not disclose that phytase can reduce the formation of deposits in heat transfer equipment in a fuel ethanol plant. A1007; *see also generally* A1580-1610 (Veit). Veit contains no examples of actually using phytase in an industrial ethanol plant, much less an example in which a reduction in the formation of deposits in a piece of heat transfer equipment was reported. A1009-11; *see also generally* A1580-1610 (Veit). Rather, Veit describes only a single laboratory experiment using a 500 ml glass bottle (about two cups of liquid). A1009, A1597-99 (Veit (16:30-18:2) (Example 1)). This experimental protocol was designed solely to evaluate enzyme and fermentation efficiency. A1009-11, A1597-98 (Veit (16:30-17:6)). There is no way to tell whether the product of the experiment in Example 1 of Veit actually would have formed a deposit had it contacted a heated surface like a heat exchanger of a fuel ethanol plant. A1011. And the results of the experiment in Example 1 of Veit

provide no information about whether a phytase, if actually used in an actual fuel ethanol plant, would necessarily or inevitably result in reduced deposit formation in downstream processing heat transfer equipment. A1011-12.

b. Practice of Veit will not *necessarily* result in deposition reduction.

The disclosure of Veit easily can be practiced in a manner that would improve fermentation (i.e., by improving amylase action) per the intent of Veit, and yet *not* result in U.S. Water's claimed reduction in the formation of phytic acid deposits. A1007-15, A760-62, A863-66. Veit provides no guidance for the person skilled in the art regarding what phytases to use, no guidance regarding what concentrations to employ, and no guidance with respect to any of the other critical reaction conditions that could result in inevitable and invariable reduction in the formation of insoluble deposits of phytic acid in the heat transfer equipment of a fuel ethanol processing plant. A1007-15, A1588-89 (Veit (7:30-8:15)). Indeed, it is only with the benefit of the knowledge from U.S. Water's patents that one of ordinary skill would even know that a reduction in the formation of deposits could be accomplished, much less perform the routine experimentation necessary for a given phytase to find the concentration, addition point, residence time, and parameters that would result in a reduction in the formation of deposits. *See* A991-93, A1007, A1012-13, A1015, A1021-26.

The record is clear that a number of variables, such as solubility, rate of release of phytic acid from starch, temperature, and characteristics of different phytase enzymes, affect the formation of phytic acid deposits. A1113-14, A754-62. The kinetics of phytic acid deposition also means that one easily could add enough phytase to accomplish the purpose of Veit and yet never achieve a reduction in the formation of deposits because the amount of phytic acid remaining in solution would result in the same amount of deposit formation. A850-60. Thus, as established by competent expert opinion fully on record, “the lack of guidance provided in Veit means it is highly unlikely that one skilled in the art would ever have chosen a combination of phytase, concentration, addition point(s) and reaction conditions that would have led to a reduction in the formation of deposits.” A1012-14, A760-62, A863-66. Regardless, practicing Veit will not “necessarily and inevitably” lead to the reduction of deposits in a fuel ethanol plant any more than using the ingredients in a Betty Crocker cake box without following the recipe will result in a cake.

3. The Antrim reference.

Like Veit, the Antrim reference is not directed to deposit reduction, does not provide guidance on conditions that are necessary for deposit reduction, and will not inevitably result in deposit reduction.

a. The Antrim reference is not directed to deposit reduction.

U.S. Patent No. 5,756,714 to Antrim et al. ("Antrim"), entitled "Method for Liquefying Starch," was issued on May 26, 1998. A1612-23 (Antrim). Antrim is directed to improvements in the liquefaction of corn starch, which occurs well before fermentation and distillation of ethanol processing fluid. A1021-22 (*see especially* ¶ 391), A1615 (Antrim (4:5-21 and 4:35-37)). Specifically, it identifies phytic acid as an "enzyme inhibiting composition" present in starch and methods of using a phytase enzyme to "reliev[e] phytate-based inhibition of amylase action." A1021-22, A1615 (Antrim (4:9-13, 4:35-37, 4:44-59)). Antrim does not mention the distillation process, much less that phytic acid deposits form in heat transfer equipment during that process. A1021-22.

b. Antrim provides no data from a fuel ethanol plant that could evidence deposit reduction.

Like Veit, Antrim provides no data from a commercial fuel ethanol plant that could evidence a reduction in deposits; rather, the only data in Antrim are from small bench-top experiments. A1023. Virtually all of the bench-top experiments in Antrim use starch from a corn wet milling process, which contains relatively little phytic acid. A1022-23, A1618 (Antrim (10:13-18)). While Example 8 uses corn, it merely shows that phytic acid can be extracted from ground whole corn using room temperature water. A1022, A1619-20 (Antrim (12:55-13:45)). Antrim provides no examples of using phytase to reduce the formation of deposits in fuel ethanol production. *See* A1022.

c. Antrim provides a wide range of conditions that do not inevitably result in deposition reduction.

According to Antrim, phytase may be derived from any number of "phytase producing microorganisms," including bacteria and filamentous fungi and yeasts. A1023-24, A1616 (Antrim (6:52-56)). Antrim provides wide ranges of temperature (up to 110°C), pH (4-6), and dosage (0.1 to about 100 units of phytase per gram of starch). A1023-24, A1617 (Antrim (7:6-11 and 7:39-65)). And the designated pH ranges in Antrim are chosen to optimize the alpha-amylase enzyme activity—not to control deposits. A1023; *see also generally* A1612-23 (Antrim). As such, the wide range of choices for reagents and conditions in Antrim "could lead to very different results in terms of the phytic acid and phytate salts in the ethanol processing fluid." A1023.

Antrim fails to provide any guidance with respect to the many variables that affect the formation of deposits containing phytic acid and phytates. A1023-24. Because of the massive amount of phytic acid present in a fuel ethanol plant and the phenomenon of zero-order kinetics, even if Antrim reduced some amount of phytic acid in the ethanol processing fluid, it would not "necessarily" be sufficient to reduce deposit formation in downstream ethanol processing fluid. A1024, A850-60, A760-62. In fact, it is known that adding too little phytase, or adding phytase to liquefaction, does not reduce deposit formation. A389 (Young Dep. at 94:20-96:2). And fuel ethanol plants that used the Genencor Maxaliq® product, which contained phytase, have in fact continued to experience fouling. A1826. Thus, following the teaching of Antrim does not inevitably and invariably lead to a reduction in deposit formation in a fuel ethanol processing plant. A1021-27.

Antrim provides a wide range of variables for achieving Antrim's stated purpose of improving liquefaction. A1021-27. And as with Veit, it is only with the benefit of the knowledge from U.S. Water's patents that one of ordinary skill would even know that a reduction in the formation of deposits could actually be accomplished by picking and choosing from among Antrim's variables and actually perform the routine experimentation necessary for a given phytase to find the concentration, addition point, residence time, and parameters within Antrim that would result in a reduction in the formation of deposits. A1021-27.

II. This Litigation and the District Court's Decision.

On December 17, 2013, U.S. Water sued Novozymes for induced infringement of its '137 patent as a result of Novozymes' sale and offer of sale of Phytaflow® to ethanol plants for deposit control. A225-32. U.S. Water subsequently asserted infringement of its '399 patent on the same grounds. A153-

62. In June 2014, Novozymes filed counterclaims for declaratory judgment of non-infringement and invalidity of the patents-in-suit. A191-95.[5]

The parties filed cross-motions for summary judgment. A1341-43, A1450-51. U.S. Water moved for summary judgment on a variety of claims and defenses, including direct infringement and no inequitable conduct. A1341-43. Novozymes moved for summary judgment on noninfringement and invalidity. A1450-51. Among other positions, Novozymes contended that the patents-in-suit were invalid as inherently anticipated by the prior art Veit and Antrim references. A1529-36.

In its opposition to Novozymes' motion, U.S. Water chronicled extensive evidence that both Veit and Antrim could be practiced in a manner that did not result in deposit reduction. A1733-50 (S.J. Opp'n at 74-91), A28 (Order at 27). That is, U.S. Water did not dispute that if, in hindsight and with benefit of the disclosure of the U.S. Water patents, the correct combination of parameters from the various ranges of the various conditions, such as pH, temperature, and dosage disclosed in Veit or Antrim were selected, Veit or Antrim *might* result in deposit reduction. A1748-50 (S.J. Opp'n at 89-91). Rather, U.S. Water disputed that deposit reduction was *necessarily*, i.e., inevitably and invariably, the result of practicing either reference. A1748-50 (S.J. Opp'n at 89-91). Indeed, "[a]n infinite

[5] Novozymes thereafter amended its answer to add a counterclaim of inequitable conduct and the defense of collateral estoppel. A248-67 (Countercl. ¶¶ 45-98), A551-52 (Countercl. ¶ 11).

number of monkeys at an infinite number of typewriters for an infinite amount of time may eventually write Hamlet. This does not mean that a single monkey equipped with the same typewriter *necessarily* would. So too here. The record is clear that it is *not* inevitable that one of skill in the art practicing Veit would *necessarily* choose the various parameters Novozymes instructs its customers to follow." A1749 (S.J. Opp'n at 90).

On July 29, 2015, the district court granted Novozymes' motion for summary judgment[6] that the asserted claims of the patents-in-suit were invalid as inherently anticipated by the prior art. A2-37 (Order); *see also U.S. Water Services, Inc. v. Novozymes A/S*, __ F. Supp. 3d __, No. 13-cv-864-jdp, 2015 WL 463452 (W.D. Wis. July 29, 2015). Crediting U.S. Water's evidence, the district court found that "U.S. Water has adduced evidence … that practicing Veit and Antrim will not *always* result in deposit reduction." A28 (Order at 27) (emphasis in original). The district court nonetheless found that summary judgment was "appropriate because the evidence of record establishes that deposit reduction is a natural result of the methods for adding phytase during ethanol production that Veit and Antrim disclose." A26-27 (*Id.* at 25-26).

[6] The court also correctly granted U.S. Water's motion for summary judgment of no inequitable conduct, which is the subject of Novozymes' cross appeal.

First, the district court found it undisputed that both patents-in-suit and the prior art disclose the chemical process through which phytase breaks down phytic acid, and that the ranges of various conditions in the prior art overlapped with the ranges of the various conditions disclosed in the asserted patents. A18, A27 (*Id.* at 17, 26). The court thus determined that, while U.S. Water had proffered evidence that, because of the many variables that affect deposit reduction, merely adding phytase at some stage in the production process would not necessarily reduce phytic acid deposits, for purposes of inherency, it was sufficient that the prior art disclosed a known chemical reaction and overlapping ranges of conditions. *See* A18, A27 (*Id.* at 17, 26).

Second, the district court characterized U.S. Water's opposition as arguing only that Veit and Antrim did not inherently anticipate because the two references had a different *purpose* than the patents-in-suit and because a skilled artisan would not appreciate that Antrim or Veit would reduce deposits, and held that the "differences between the goals of Veit and Antrim and the goals of the patents-in-suit do not preclude the former from anticipating the latter." A28 (*Id.* at 27). That is, since the patents-in-suit "teach using phytase during ethanol production, in the same dosages and under the same conditions, to perform the same chemical reaction," the court reasoned that Veit and Antrim inherently disclose that benefit, "[e]ven though [they] do not expressly identify the benefit that U.S. Water's patents identify." A28 (*Id.*).

Third, the district court sought to distinguish Federal Circuit precedent holding that if teachings of the prior art can be practiced in a way that lacks the allegedly inherent property, the prior art does not inherently anticipate. A29-32 (*Id.* at 28-31). The court noted that these cases addressed composition (as opposed to method) claims. A31 (*Id.* at 30). Thus, while the court reasoned that a "results-orientated analysis makes sense" in the context of composition claims, according to the court such logic was inapplicable here, where method claims involve the use of a known chemical reaction, even if directed to a new benefit. *Id.* (citing *In re Armodafinil Patent Litig.*, 939 F. Supp. 2d 456, 465 (D. Del. 2013); *Glaxo Inc. v. Novopharm Ltd.*, 52 F.3d 1043, 1045 (Fed. Cir. 1995)). And, while the court considered precedent holding that prior art methods that only sometimes result in the claimed method do not inherently anticipate, the court also found such cases distinguishable again on the basis that (according to the district court) U.S. Water had merely "identif[ied] a new 'goal' for the phytase process disclosed by a prior art reference." A29-30 (Order at 28-29 (discussing *MEHL/Biophile Int'l Corp. v. Milgraum*, 192 F.3d 1362 (Fed. Cir. 1999)).

Finally, the district court retreated to the old axiom "'that which would literally infringe if later anticipates if earlier.'" A32 (Order at 31 (quoting *Bristol-*

Myers Squib Co. v. Ben Venue Labs., Inc., 246 F.3d 1368, 1378 (Fed. Cir. 2001))). In particular, the court concluded that the asserted claims were inherently anticipated because, if plants practicing Veit or Antrim happened to "stumble upon a reduction in deposits[,] U.S. Water's patents, as asserted, would expose the plant to liability for infringement." A32 (*Id.* at 31). The district court claimed (without citation to the record) that this was "the very position U.S. Water ha[d] taken in this case, alleging that several of Novozymes's customers, *who practice Veit and Antrim*, have infringed on the patents-in-suit because they experienced a reduction in deposits." *Id.* (emphasis added).[7] And, in so claiming, the court found that, since "U.S. Water [had] not persuasively explain[ed] how the public could continue to practice Veit and Antrim if the patents-in-suit are valid … they anticipated the invention on which U.S. Water now claims that Novozymes and its customers have infringed." A32 (*Id.*).

SUMMARY OF ARGUMENT

The district court erred when it granted summary judgment of inherent anticipation because there is an acknowledged, material factual dispute. Namely, inherent anticipation may be found *only* where the allegedly inherent limitation is

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[7] U.S. Water has made no such accusation. The plants it identified are users of Novozymes' Phytaflow® product, which is marketed and used expressly to reduce fouling. *See* A1722-56, A1355-56, A765, A771; *see also infra*, Argument, Part III.B.3. Indeed, it is unclear whether *any* plants practice either Veit or Antrim, let alone that the Novozymes' customers at issue in this litigation do.

inevitably and necessarily present in the prior art. *See, e.g.*, *MEHL/Biophile Int'l Corp.*, 192 F.3d at 1365; *see also infra*, Argument, Part II. Here, the district court recognized that U.S. Water had adduced evidence that the prior art will not inevitably reduce deposits. A28 (Order at 27). This was enough to require the denial of Novozymes' motion. Instead, the district court committed a series of legal and factual errors, which resulted in an erroneous grant of summary judgment.

First, the district court brushed aside U.S. Water's evidence that the prior art would not always result in deposit reduction on the ground that deposit reduction is merely a new goal or benefit of the invention. A28 (Order at 27). But deposit reduction in a fuel ethanol plant is the *sine qua non* of this invention—it was what was discovered—and, accordingly, it is an express limitation in the asserted claims. *See infra*, Argument, Part III.A & III.B.1.

Second, the district court found that, because the ranges in the U.S. Water patents of, for example, pH, temperature, and dosage, have some overlap with ranges found in the prior art, inherency is satisfied. In the district court's view, it is enough that each condition in the challenged patents falls somewhere within the many broad ranges disclosed in the prior art. A18-23, A32 (Order at 17-22, 31). But, regardless of whether, as the district court found, there is range overlap, inherency still requires a showing of inevitability. And here, given the number of variables that impact whether deposit reduction is achieved, and given the wide ranges disclosed in the prior art, there is ample evidence that the prior art will not invariably result in the claimed invention. *See infra*, Argument, Part III.B.2.

Third, the district court concluded that, because one practicing the prior art might "stumble" on deposit reduction, and thereby potentially infringe the asserted claims, the prior art inherently anticipates the U.S. Water patents. A32 (Order at 31). Setting aside that this conclusion centers on a clear record error—namely, that U.S. Water has accused plants that merely practice the prior art of infringement (it has not)—it also turns on a clear error of law: "practicing the prior art" is not a defense to infringement, as detailed below. *See infra*, Argument, Part III.B.3.

ARGUMENT

The prior art at issue in this case is directed solely toward using phytase to improve liquefaction, saccharification, or fermentation. The prior art admittedly does not disclose the use of phytase for deposit reduction. Thus, the burden is on Novozymes to prove by clear and convincing evidence that practicing the prior art methods—directed to a wholly different purpose—will inevitably and always meet the limitation of U.S. Water's patents, which require a reduction in the formation of deposits. But as the district court recognized, Veit and Antrim will not *always* result in deposit reduction.[8] As such, Veit and Antrim cannot, as a matter of law, inherently anticipate U.S. Water's claimed inventions, which require deposit reduction.

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[8] Respectfully, this statement, although sufficient to identify an issue of fact, understates the force of U.S. Water's record. More accurately, the evidence shows that Veit and Antrim *never* will result in deposit reduction. Indeed, there is no

I. Standard of Review.

This Court reviews *de novo* grants of summary judgment based on 35 U.S.C. § 102 anticipation. *Crown Packaging Tech., Inc. v. Ball Metal Beverage Container Corp.*, 635 F.3d 1373, 1383 (Fed. Cir. 2011) (citing *Telemac Cellular Corp. v. Topp Telecom, Inc.*, 247 F.3d 1316, 1327 (Fed. Cir. 2001)) (reversing summary judgment of inherent anticipation where conflicting expert testimony created a disputed factual issue). In other words, "[t]his court reviews a district court's grant of summary judgment by reapplying the standard applicable at the district court." *MEHL/Biophile Int'l Corp.*, 192 F.3d at 1365 (citing *Conroy v. Reebok Int'l, Ltd.*, 14 F.3d 1570, 1575 (Fed. Cir. 1994)). "Summary judgment is appropriate only when "'there is no genuine issue as to any material fact and ... the moving party is entitled to a judgment as a matter of law.'" *Id.* (quoting Fed. R. Civ. P. 56(c)). "In its review, this court draws all reasonable inferences in favor of the non-movant." *Id.* (citing *Anderson v. Liberty Lobby, Inc.*, 477 U.S. 242, 255 (1986)).

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evidence on this record that any person at any time ever practiced Veit or Antrim and achieved deposit reduction.

II. To Inherently Anticipate, Novozymes Must Prove by Clear and Convincing Evidence that Veit and Antrim Inevitably and Necessarily Accomplish U.S. Water's Claimed Deposit Reduction.

A prior art reference may anticipate when a claim limitation that is not expressly found in the four corners of the reference is nonetheless inherent. *See, e.g.*, *In re Oelrich*, 666 F.2d 578, 581 (C.C.P.A. 1981). But, under clear precedent, such inherent anticipation occurs *only* where there is clear and convincing evidence that the limitation is inevitably present in the prior art reference. *See, e.g.*, *MEHL/Biophile Int'l Corp.*, 192 F.3d at 1365; *Finnigan Corp. v. ITC*, 180 F.3d 1354, 1366 (Fed. Cir. 1999). Simply put, inherency requires inevitability and invariability. *See In re Oelrich*, 666 F.2d at 581. It cannot be established by mere "'probabilities or possibilities.'" *Id.* (quoting *Hansgirg v. Kemmer*, 102 F.2d 212, 214 (C.C.P.A. 1939)); *see also Trintec Indus., Inc. v. Top-U.S.A. Corp.*, 295 F.3d 1292, 1295 (Fed. Cir. 2002) ("Inherent anticipation requires that the missing descriptive material is 'necessarily present,' not merely probably or possibly present, in the prior art."); *Finnigan Corp.*, 180 F.3d at 1366 ("The mere possibility that Figure 2 might be understood by one of skill in the art to disclose nonresonance ejection is insufficient to show that it is inherently disclosed therein."); *Glaxo*, 52 F.3d at 1047-48 (no inherency where defendant's experts reproduced a prior art method "thirteen times and each time they made [the claimed] crystals," because the patentee's chemists produced different crystals from the same method). "The mere fact that a certain thing *may* result … is not sufficient" (*In re Oelrich*, 666 F.2d at 581; *see also Hansgirg*, 102 F.2d at 214), because "[o]ccasional results are not inherent." *MEHL/Biophile Int'l Corp.*, 192 F.3d at 1365; *see also In re Montgomery*, 677 F.3d 1375, 1384 (Fed. Cir. 2012) (Lourie, J., dissenting) ("The keystone of the inherency doctrine is inevitability. … Our precedent has been steadfast in this strict requirement of inevitability. … Absent inevitability, inherency does not follow even from a very high likelihood that a prior art method will result in the claimed invention.") (note that the majority in *Montgomery* agreed with the dissent's statement of law (*id.* at 1381)). The reference as taught must *always*—not merely sometimes—result in the patented invention. *See Trintec Indus., Inc.*, 295 F.3d at 1295. It follows that, if the teachings of the prior art can be practiced in a way that does not result in the allegedly inherent limitation, the prior art in question does not inherently anticipate. *Glaxo*, 52 F.3d at 1047-48 (district court correctly found that process that yielded claimed compound most, but not all, of the time did not inherently anticipate).

The requirement of inevitability is well-illustrated in *MEHL/Biophile International Corp. v. Milgraum*. *See* 192 F.3d 1362. In *MEHL*, this Court found that claims directed to a method of laser hair removal wherein the laser was vertically aligned over the hair follicle were not inherently anticipated by a prior art laser manual teaching the use of a laser to remove tattoos. While both methods used the same type of laser on human skin, and both *could* result in vertical alignment of the laser over the hair follicle (and reduction in hair), the manual did not expressly disclose vertically aligning the laser over the hair follicles. *See id.* at 1364-66. Thus, while it was possible (if not likely) that the laser operator *could* align the laser over the follicle (and thus remove hair), it was not inevitable. *See id.* at 1365-66. And "[t]he possibility of such an alignment does not legally suffice to show anticipation. Occasional results are not inherent." *Id.* at 1365 (citing *In re Oelrich*, 666 F.2d at 581).

In contrast to the laser manual, a second reference, the Polla Article, *did* inherently anticipate the claims. *Id.* at 1366. This was because the Polla Article expressly noted the alignment of the laser over the hair follicle, as claimed in the asserted patents. *See id.* There was thus nothing left to chance: the "natural result" of the Polla Article was aligning the laser over the hair follicle and the removal of hair. *See id.* To be sure, one of ordinary skill may not have had that goal in mind or even have been aware that hair loss occurred. *See id.* But, given that the Polla Article was clear that vertical alignment occurred, this was of no moment. In sharp contrast to the *occasional* results of the product manual, the "natural result" of following the Polla Article was to practice the claimed invention. *See id.* at 1366-67.

Glaxo Inc. v. Novopharm Ltd. is also instructive. 52 F.3d at 1047-48. In *Glaxo*, this Court affirmed a finding of the district court that patents directed to a specific crystalline form of the compound ranitidine hydrochloride (Zantac®) were not inherently anticipated by an example in a prior art patent where following that example did not *invariably* yield the claimed highly crystalline form. *Id.* Indeed, Novopharm's experts performed the prior art example thirteen times and obtained the claimed crystalline form each time, but inherent anticipation was not present because Glaxo's own experts followed the example and did not obtain the claimed form. *Id.* at 1047; *see also Glaxo, Inc. v. Novopharm Ltd.*, 830 F. Supp. 871, 877 (E.D.N.C. 1993), *aff'd*, 52 F.3d 1043 (Fed. Cir. 1995). And while the parties disputed whether the other had correctly followed the prior art example's teachings, because there was evidence that the example *could be* practiced in a manner that did not invariably result in the claimed compound, inherency was not present. *See* 52 F.3d at 1048; *see also Glaxo, Inc.*, 830 F. Supp. at 877 ("The mere fact that certain things may result from a given set of circumstances is not sufficient.") (citing Rosenberg, Patent Law Fundamentals § 7.04).

III. The District Court's Failure to Follow Long-Standing Federal Circuit Precedent Requires Reversal of Its Summary Judgment Ruling.

A. The district court strayed from the bedrock principle that inherency requires inevitability and invariability.

The district court committed several errors in concluding that Veit and Antrim inherently anticipate the asserted claims. First and foremost, however, the court strayed from the one fundamental, overriding, and threshold requirement of inherent anticipation, i.e., that following the disclosure of the prior art must necessarily—meaning, *inevitably*—result in all limitations of the claimed invention. Indeed, the word "inevitable" appears nowhere in the Order. Only by failing to follow this immutable tenet of patent law could the court possibly find inherent anticipation while at the same time expressly acknowledging that "U.S. Water has adduced evidence that … practicing Veit and Antrim will not *always* result in deposit reduction." A28 (Order at 27 (emphasis in original)). This basic error, standing alone, is sufficient to warrant reversal.

B. The district court's rationales for finding inherency were flawed.

The district court nonetheless offered at least three principal reasons for its conclusion that the asserted claims are inherently anticipated. All are flawed.

1. Deposit reduction is not merely a *goal* or *benefit* of the invention; it is an express limitation of all asserted claims.

First, the district court reasoned that "the differences between the goals of Veit and Antrim and the goals of the patents-in-suit do not preclude the former from anticipating the latter." A28 (Order at 27) (citing *Leggett & Platt, Inc. v. VUTEk, Inc.*, 537 F.3d 1349, 1356 (Fed. Cir. 2008)). The court thus reasoned that U.S. Water's evidence that Veit and Antrim do not inevitably result in deposit reduction was "irrelevant." *Id.*

This reasoning overlooks both the asserted claims and governing law. To be sure, this Court's precedent does hold that a new goal or benefit, on its own, will not salvage an otherwise anticipated claim where the prior art inevitably practiced all limitations of the invention. *See MEHL/Biophile Int'l Corp.*, 192 F.3d 1362. That is not the case here. Contrary to the understanding of the district court, deposit reduction is not merely a goal or benefit of the claimed methods: it is an express limitation of all claims. *See* A141-42 ('137 patent (all claims)), A150-52 ('399 patent (all claims)). And once one understands that the patents-in-suit *require* deposit reduction, it readily follows that summary judgment is improper unless the prior art undisputedly, and inevitably reduced deposits. Here, as detailed above (and as found by the district court), that is not the case. A28 (Order at 27).

2. The district court's *per se* range rule is based on a misunderstanding of both *Atlas Powder* and *Perricone*.

a. In *Atlas Powder,* the Federal Circuit correctly applied the "inevitability" test of inherent anticipation.

Second, the district court misapplied this Court's decisions in *Atlas Powder Co. v. IRECO, Inc.*, and *Perricone v. Medicis Pharmaceutical Corp.*, to find that the patents-in-suit are inherently anticipated simply because the ranges disclosed in Veit and Antrim overlap with the ranges disclosed in the patents. *See* A18 (Order at 17). In particular, the district court relied on language from *Atlas Powder* relating to chemical composition claims: "[W]hen a patent claims *a chemical composition* in terms of ranges of elements, any single prior art reference that falls within each of the ranges anticipates the claim." A18 (Order at 17 (emphasis added) (quoting *Atlas Powder Co. v. IRECO, Inc.*, 190 F.3d 1342, 1346 (Fed. Cir. 1999) and citing *Perricone v. Medicis Pharm. Corp.*, 432 F.3d 1368, 1377 (Fed. Cir. 2005))). Thus, based on its view of *Atlas Powder*, and on its view that deposition reduction was merely a goal or benefit of the invention, the district court reasoned that "it is enough for Veit or Antrim to expressly disclose values or ranges of values that fall within the ranges contained in the patents-in-suit. The ranges do not need to precisely overlap, nor does the prior art's range need to fit entirely within the range claimed by the patents-in-suit." A18 (Order at 17). But the question presented in *Atlas Powder* was distinct from the issue here. And in fact, as seen below, it was the district court's misreading of *Atlas Powder* that apparently led it astray in this case.

By way of background, the *Atlas Powder* patent claimed explosive compositions that combined a particular percentage of blasting composition with a particular percentage of water-in-oil emulsion, e.g., a combination of 60%-90% solid oxidizer salt with 10%-40% water-in-oil emulsion. *See* 190 F.3d at 1344. Unlike the *Atlas Powder* patents, U.S. Water's patents do not claim chemical compositions, much less chemical compositions in terms of ranges: they are method claims that recite various steps for the reduction of phytic acid deposits. *See generally* A141-42 ('137 patent (all claims)), A150-52 ('399 patent (all claims)). Simply put, the district court misinterpreted, and thus misapplied, the holding of *Atlas Powder* to the facts of this case was inapt.

In *Atlas Powder*, after determining whether the prior art ranges and those of the claimed composition overlapped, this Court nonetheless still *required* that the asserted prior art inevitability and invariably produce a result that met every limitation. *See Atlas Powder Co.*, 190 F.3d at 1344-48. In other words, the *Atlas Powder* court's analysis and determination did not rest on whether the prior art compound fell (or did not fall) within certain ranges. *See id.* That was only one step in the analysis. *See id.* After taking this step, the Court then also expressly determined—by a detailed analysis of the evidence of record—that the asserted prior art always resulted in the claimed compound. *See id.* And because the detailed evidence showed that it did, the Federal Circuit found that inherency was present. *See id.*

To illustrate, in *Atlas Powder*, this Court first observed that the prior art (specifically, Egly and Butterworth) expressly disclosed all but one element of the Atlas Powder claims: "sufficient aeration [be] entrapped to enhance sensitivity to a substantial degree." *Id.* at 1346. That is, the make-up of the compositions found in the prior art and the claimed compositions overlapped, except that the prior art was not express that the compositions had "sufficient aeration … entrapped to enhance sensitivity to a substantial degree." *See id.*

Next, as to the missing element, this Court applied the "inevitability" test to determine whether that missing claim element was inherently found in the prior art compositions. *Id.* at 1348 ("The other experts agreed that the emulsions described in both Egly and Butterworth would inevitably and inherently have interstitial air remaining in the mixture."); *id.* ("These tests, therefore, support the finding that 'the emulsions described by Butterworth, combined with the ratios of ANFO disclosed by Butterworth, would inevitably and inherently have interstitial air remaining up to approximately 40% emulsion.'"); *id.* ("These tests, therefore, further support the court's finding that 'emulsions described in the Egly Patent, combined with either AN or ANFO, would inevitably and inherently have interstitial air remaining in the mixture up to approximately 40% emulsion to 60% solid constituent.'"). And, only after concluding that the missing limitation was inevitably present based on "exhaustive evidence" that compositions in the prior art ranges would invariably include "sufficient aeration … to enhance sensitivity," did this Court find inherency. *Id.*; *see also id.* at 1349 ("While Egly compositions containing amounts approaching 67% by weight of water-in-oil emulsions may have little or no entrapped air, the evidence established that at emulsion levels below 40%, Egly compositions 'inevitably and inherently' trap sufficient amounts of air to enhance sensitivity.").

Three aspects of this Court's analytical approach in *Atlas Powder* warrant further attention. First, this Court started by examining *every* limitation of the claims at issue. *See id.* at 1346. Indeed, it considered which limitations were and were not expressly disclosed in the art. *See id.* Second, having identified the limitation that was not expressly disclosed, this Court then determined whether that missing limitation was "*inevitably*" present in the prior art compositions. *Id.* at 1347-49 (emphasis added). And, third, in order to reach its conclusion, this Court examined the evidence of inevitability, including "substantial amounts of expert testimony" and "data showing extensive testing" of the prior art compositions. *Id.* at 1348-49. In other words, this Court recognized and respected the intensely factual nature of the inquiry into inevitability.

Given the above, it is apparent that the *Atlas Powder* Court did not announce a *per se* "overlap" rule—one that this Court intended to trump the inevitability inquiry. The case does not create a rigid test that, regardless of the context of the claims, the prior art, and the evidence, claims will be inherently anticipated based merely on a mechanical comparison of the ranges in the prior art to the asserted claims.

Here, however, the district court bypassed the required, careful inquiry into inevitability. Indeed, by misreading *Atlas Powder* as establishing a hard-and-fast "overlap" rule, the district court ran afoul of this Court's precedent that inherency requires clear evidence of inevitability of each limitation that is not express in the prior art. This was error. And under clear precedent, including *Atlas Powder*, the Court need look no further than the district court's recognition that Veit and Antrim will not inevitably result in deposit reduction. A28 (Order at 27). This was sufficient to defeat summary judgment.

b. The nature of the invention and prior art in this case differs greatly from those in *Atlas Powder*.

Moreover, the distinction between *Atlas Powder* and the present case also extends to the nature of the inventions and the prior art at issue. As noted above, the invention in *Atlas Powder* was a composition, i.e., a collection of ingredients. 190 F.3d at 1344. The prior art disclosed the same collection of ingredients in the same amounts, albeit expressed in over lapping ranges. *See id.* In contrast, the

U.S. Water inventions are drawn to *processes* that comprise multiple steps, the results of which can be affected by numerous slight variations in how the steps are carried out as well as significant variations in the parameters in the fuel ethanol plant and ethanol processing fluids themselves. *See supra*, Statement of the Case, Part I.D-E. That these various combinations may be randomly patch-worked together from the broad ranges in the prior art does not mean that the prior art would *always* result in deposit reduction. And the record is clear that it would not.

Indeed, the prior art in this case involves processes that can be practiced over an extraordinarily wide range of parameters. For example, Veit contains only a short passage that discloses how much phytase can be added to achieve improved fermentation. *See* A1589 (Veit (8:11-15)). The low end of Veit's range is 0.005 FYT/gram DS (phytase units of activity per gram of dissolved solids). *Id.* This amount is so low that no one has contended it could achieve U.S. Water's claimed reduction in the formation of deposits—indeed, Novozymes' expert has not opined that such a low amount could or would result in deposit reduction. And yet, one of ordinary skill would be squarely within the literal teaching of Veit if he or she added a phytase in the amount of 0.005 FYT/gram DS. The same is true of Antrim, which discloses the use of a phytase concentration as low as 0.1 phytase units per gram of starch. A1617 (Antrim (7:6-11)).

Similarly, Veit discloses other broad ranges that could be employed for carrying out the steps it describes. Fermentation can be from twenty-four to ninety-six hours, and can be carried out at a temperature between 24°C and 34°C and at a pH of from three to six. A1585 (Veit (4:5-9)). The phytase can be selected from one of the many phytases described at page 8, line 21 through page 9, line 25. *See* A1589-90 (Veit (8:21-9:25)). The phytase can be added either during saccharification or fermentation, or during both. A1582 (Veit (1:7-8)).

These enormous variations in the process steps and parameters, as well as wide variations of the phytases and amounts of phytase, are all within the scope of Veit's disclosure—and are all left to the picking and choosing of skilled artisans. The result is that, (i) one could actually practice the disclosures of Veit and Antrim in any of a million different ways and combinations of parameters, (ii) no two skilled artisans might ever practice Veit or Antrim in the same way, and (iii) one easily could practice Veit and Antrim and not achieve a reduction in the formation of deposits. *See supra*, Statement of the Case, Part I.E.; *see also* A28 (Order at 27) (noting evidence that the prior art will not always result in deposit reduction).

c. The district court similarly misapplied *Perricone*.

The district court sought to bolster its reliance on *Atlas Powder* with further citation to *Perricone v. Medicis Pharmaceutical Corp.* A18, A22, A29 (Order at 17, 21 28) (citing 432 F.3d at 1376-77). The court's analysis, however, again missed the real import of *Perricone*, which addressed two different groups of method claims. The first group of method claims recited the application of a particular composition to treat skin to achieve certain benefits. The second recited applying the same composition to treat sunburned skin. 432 F.3d at 1378. The prior art (Pereira) disclosed applying a substantially identical composition to normal skin, albeit for a different purpose. *Id.* at 1377 ("[a]s the district court correctly noted, Pereira's range entirely encompasses, and does not significantly deviate from, Dr. Perricone's claimed ranges").

As in *Atlas Powder*, the *Perricone* Court again applied the inevitability test in determining inherent anticipation. *Id.* at 1378 ("when considering a prior art method, the anticipation doctrine examines the natural and inherent results in that method"). This Court held the first group of claims was inherently anticipated by Pereira—an unremarkable result given that there was no dispute that Pereira disclosed the very same method and would inevitably achieve the very same benefit as in the first group of claims. *Id.* ("[i]f Pereira discloses the very same methods, then the particular benefits must naturally flow from those methods even if not recognized as benefits at the time of Pereira's disclosure").

Importantly, however, this Court *reversed* the district court's holding of inherent anticipation on the second group of claims directed to a method of using the compound disclosed by Pereira to treat skin sunburn. *Id.* at 1378-79. The district court's anticipation analysis was "flawed" because Pereira, while disclosing "topical application," did not suggest application of Pereira's lotion to skin sunburn. *Id.* at 1379. "In other words, the district court's inherency analysis goes astray because it assumes what Pereira neither disclosed nor rendered inherent." *Id.* To hold otherwise would be to improperly "ignore" or "dismiss" the explicit language of Dr. Perricone's claimed method, "applying to the skin sunburn." *Id.* (application to skin surfaces generally is not analogous to skin sunburn; "unrealized possibilities do not alter the analysis" where the prior art does not disclose an express limitation).

So too here. The district court ignored or dismissed the explicit language of U.S. Water's claimed method, which requires "reduction in the formation of deposits." Because this limitation is neither expressly disclosed nor inherently (i.e., always inevitably and invariably) present in either Veit or Antrim, such claims cannot be anticipated by those references.

3. The district court's reliance on the discredited doctrine of "practicing the prior art" is a further indication of error.

The district court also based its decision on the notion that the U.S. Water patents, if valid, would prevent the public from practicing Veit and Antrim:

> Ultimately, the doctrine of inherent anticipation enforces the "basic principle" of patent law that "[t]he public remains free to make, use, or sell prior art compositions or processes, regardless of whether or not they

> understand their complete makeup or the underlying scientific principles which allow them to operate." The patents-in-suit, if valid, would prevent the public from practicing Veit and Antrim. … U.S. Water does not persuasively explain how the public could continue to practice Veit and Antrim if the patents-in-suit are valid.

A31-32 (Order at 30-31 (quoting *Atlas Powder Co.*, 190 F.3d at 1348)).

This too was error. The concept of "practicing the prior art" as a defense to infringement has long been rejected by this Court. *See, e.g.*, *Cordance Corp. v. Amazon.com, Inc.*, 658 F.3d 1330, 1337 (Fed. Cir. 2011).

> A "practicing the prior art" defense typically refers to the situation where an accused infringer compares the accused infringing behavior to the prior art in an attempt to prove that its conduct is either noninfringing or the patent is invalid as anticipated because the accused conduct is simply "practicing the prior art." In Tate, this court explained that accused infringers "are not free to flout the requirement of proving invalidity by clear and convincing evidence by asserting a 'practicing the prior art' defense to literal infringement under the less stringent preponderance of evidence standard." Instead "[a]nticipation requires a showing that each element of the claim at issue, properly construed, is found in a single prior art reference. 'It is the presence of the prior art and its relationship to the claim language that matters for invalidity.'"

Id. (quoting *Tate Access Floors, Inc. v. Interface Architectural Res., Inc.*, 279 F.3d 1357, 1367 (Fed. Cir. 2002) & *Zenith Elecs. Corp. v. PDI Commc'n Sys., Inc.*, 522 F.3d 1348, 1363 (Fed. Cir. 2008)); *see also Tate Access Floors, Inc.,* 279 F.3d at 1365 ("This court made unequivocally clear in [*Baxter Healthcare Corp. v.*

Spectramed, Inc., 49 F.3d 1575, 1583 (Fed. Cir. 1995)] that there is no "practicing the prior art" defense to literal infringement. … We held that substantial evidence supported a finding of literal infringement notwithstanding Spectramed's argument that its accused devices could not infringe because they were constructed using only the teachings contained in the prior art.").

The bottom line is that the validity of U.S. Water's claims at this juncture may be determined solely by an analysis of whether the prior art inherently anticipates the U.S. Water claims by clear and convincing evidence consistent with this Court's jurisprudence. As discussed above, this jurisprudence provides an unalterable framework for the analysis of inherent anticipation. It is irrelevant to the validity of U.S. Water's patents whether one could, either by chance or with the benefit of U.S. Water's patent disclosure, pick and choose from myriad possibilities that Veit and Antrim provide and achieve a reduction in the formation of deposits.

The district court conceded it was concerned with precisely such a possibility:

> U.S. Water emphasizes that the key component of its patents is deposit reduction, and that it is unlikely that anyone practicing Veit or Antrim would "stumble on" this result. But what if an ethanol plant *did* stumble upon a reduction in deposits? U.S. Water's patents, as asserted, would expose the plant to liability for infringement. Indeed, that is the very position that U.S. Water has taken in this case, alleging that several of Novozymes's

> customers, who practice Veit and Antrim, have infringed on the patents-in-suit because they experienced a reduction in deposits.

A32 (Order at 31) (citations omitted). Here, the district court made two errors. The first is obvious: whether a plant practicing Veit or Antrim happened to "stumble on" the conditions and parameters that would achieve a reduction in deposits is plainly irrelevant to validity. Rather, at this stage, the only question relevant to validity is whether a plant practicing Veit or Antrim would inevitably achieve a reduction in the formation of deposits, and the answer to that question is clearly "no."

The second is that, contrary to the district court's assertion that "[i]ndeed, that is the very position that U.S. Water has taken in this case, alleging that several of Novozymes's customers, who practice Veit and Antrim, have infringed on the patents-in-suit because they experienced a reduction in deposits" (A32 (Order at 31)), U.S. Water has never taken such a position. The record is devoid of any evidence whatsoever that any plants, let alone the accused plants, were, in fact, actually practicing Veit and Antrim.

By way of background, when U.S. Water introduced its patented pHytOUT® method(s) in 2009, it was the only phytase solution for treating fouling in ethanol plants. *See* A1044-45, A1400. While the pHytOUT® methods were met with initial skepticism among members of the industry,[9] once the pHytOUT® methods were commercially successful, Novozymes copied U.S. Water's methods with the [redacted] *See, e.g.*, A1049-57, A1435. Indeed, Novozymes instructed plants to use Novozymes' new Phytaflow® product in the same manner as the plants had used pHytOUT®. *See, e.g.*, A1055-57, A1440-41. And these instructions—and not a purported prior art use—are the basis for U.S. Water's accusations in this case. The district court's conclusion that U.S. Water levied its infringement allegations against users of the prior art Veit and Antrim methods, as opposed to plants using Phytaflow® with the intent to reduce deposits, is thus unsupported and inaccurate.

In the end, whether plants practicing Veit or Antrim could possibly infringe the patents-in-suit is not the relevant inquiry. The relevant test is whether Veit or Antrim *necessarily and always* result in the claimed deposit reduction. And here, the district court agreed that this issue is, at the very least, disputed.

IV. Disputed Issues of Material Fact Precluded Summary Judgment.

The district court found that "U.S. Water has adduced evidence that one of ordinary skill in the art would not think to practice Veit or Antrim in a way that reduces deposits and, moreover, that practicing Veit and Antrim will not *always*

[9] *See, e.g.*, A1048-49, A1058-60, A1824-26, A1427 (Novozymes employee stating [redacted] Indeed, Novozymes has estimated that such anti-fouling benefits save a plant $300,000 to $500,000 annually. A1368.

result in deposit reduction." A28 (Order at 27 (emphasis in original)). In fact, U.S. Water provided substantial evidence on this point, including that:

- Many of the teachings of Veit [and Antrim] (the wide ranges of enzymes, conditions and procedures for improving liquefaction and fermentation) "if actually implemented in a fuel ethanol plant, would have no effect at all on the formation of deposits in heat transfer equipment and beer columns." A1025-26.
- Even reducing a substantial amount of the phytic acid in a fuel ethanol plant can have no effect on the formation of deposits because the amount of phytic acid remaining will still be well beyond the saturation concentration and thus deposit formation will not be reduced. A850-55.
- The large excess of unprecipitated phytic acid and phytic acid salts means that even if one were to degrade a majority of the phytic acid and phytic acid salts, one would still have a solution that is fully saturated with phytic acid and phytic acid salts such that there would be no reduction in the formation of deposits. A859-60.
- "[O]ne can employ phytase enzyme according to [Antrim and Veit] … in ways that could sufficiently affect the phytic acid and phytates present to improve liquefaction and fermentation, but which would be

insufficient to have any effect on the formation of deposits in heat transfer equipment and beer columns." A1025-26.

- One could add enough phytase to accomplish the purpose of Veit and never see a reduction in the formation of deposits because one will still have so much phytic acid and phytic acid salts left in solution such that any reduction will not matter. A859-60.
- The teachings of Veit and Antrim do not necessarily result in the invention claimed in the patents in suit. A1007-15, A760-62, A863-66.

No doubt, Novozymes will contend that the addition of phytase in accordance with Veit will result in some, even if minimal, reduction in deposits because reduction to any degree is not a difficult requirement to satisfy. Even a casual unpacking of that argument, however, shows that it is riddled with factual conclusions that preclude summary judgment. To set forth just a few of these impermissible factual conclusions: Does Veit invariably result in any reduction of deposits? Is it difficult to reduce deposits even slightly? Will the teachings of Veit invariably result in even a minimal reduction? In sum, the record will show that this invention alone taught and achieved the claimed deposit reduction. Even a hindsight claim to minimal reductions in the prior art will fail when subjected to the factual scrutiny that was precluded by the District Court's premature grant of summary judgment.

CONCLUSION

Respectfully, the district court misunderstood the law of inherent anticipation. Under this Court's longstanding jurisprudence, the fact that the prior art does not always result in the reduction in the formation of deposits conclusively resolves the inherent anticipation question in U.S. Water's favor—and doubly so at the summary judgment stage where all factual disputes must be resolved in favor of U.S. Water. The district court's decision should be reversed and the case remanded.

PROOF OF SERVICE

In accordance with Federal Rule of Appellate Procedure 25 and Federal Circuit Rule 25, I certify that I caused this brief to be served via the Federal Circuit's CM/ECF system on counsel of record for the Defendants-Cross-Appellants.

I declare under penalty of perjury under the laws of the United States that the foregoing is true and correct.

Dated: November 4, 2015, at Madison, Wisconsin

/s/ Michelle M. Umberger
Michelle M. Umberger

CERTIFICATE OF COMPLIANCE

1. This brief complies with the type-volume limitation of Federal Rule of Appellate Procedure 32(a)(7)(B). The brief contains 11,925 words, excluding the portions exempted by Federal Rule of Appellate Procedure 32(a)(7)(B)(iii).

2. This brief complies with the typeface requirements of Federal Rule of Appellate Procedure 32(a)(5) and the type style requirements of Federal Rule of Appellate Procedure 32(a)(6). The brief has been prepared in a proportionally spaced typeface using Microsoft® Word 2010 and 14-point Times New Roman type.

Dated: November 2, 2015

/s/ Michelle M. Umberger
Michelle M. Umberger

Attorney for U.S. Water Services, Inc. and Roy Johnson

Addendum 1: Amended Judgment, August 20, 2015

IN THE UNITED STATES DISTRICT COURT
FOR THE WESTERN DISTRICT OF WISCONSIN

U.S. WATER SERVICES, INC. and
ROY JOHNSON,

Plaintiffs,

v.

NOVOZYMES A/S and NOVOZYMES
NORTH AMERICA, INC.,

Defendants.

AMENDED
JUDGMENT IN A CIVIL CASE

Case No. 13-cv-864-jdp

This action came before the court for consideration with District Judge James D. Peterson presiding. The issues have been considered and a decision has been rendered.

IT IS ORDERED AND ADJUDGED that judgment is entered in favor of defendants Novozymes A/S and Novozymes North America, Inc. declaring that claims 1, 6, 12, and 13 of U.S. Patent No. 8,415,137, and claims 1, 2, 5-12, 16-22, 25, 28-32, 34, and 35 of U.S. Patent No. 8,609,399 are invalid and dismissing plaintiffs U.S. Water Services, Inc.'s and Roy Johnson's infringement claims as moot.

IT IS FURTHER ORDERED AND ADJUDGED that judgment is entered in favor of plaintiffs U.S. Water Services, Inc. and Roy Johnson and against defendants Novozymes A/S and Novozymes North America, Inc. on defendants' counterclaim for declaratory judgment of unenforceability for inequitable conduct.

Approved as to form this 19TH day of August, 2015.

James D. Peterson
District Judge

by: Andrew Wiseman, Deputy Clerk
Peter Oppeneer
Clerk of Court

8/20/2015
Date

Addendum 2: Opinion and Order, July 29, 2015

IN THE UNITED STATES DISTRICT COURT
FOR THE WESTERN DISTRICT OF WISCONSIN

U.S. WATER SERVICES, INC. and
ROY JOHNSON,

Plaintiffs,

v.

NOVOZYMES A/S and NOVOZYMES
NORTH AMERICA, INC.,

Defendants.

OPINION & ORDER

13-cv-864-jdp

This is a patent infringement suit between competing suppliers of materials for fuel ethanol processing. Plaintiff U.S. Water Services, Inc., holds two patents related to a method of using an enzyme, phytase, to reduce deposits that build up in ethanol processing equipment. Plaintiffs accuse defendants Novozymes A/S and Novozymes North America, Inc. of indirectly infringing those patents by selling a phytase-based product. Both sides have moved for summary judgment on a multitude of issues. This opinion and order reaches only two issues, which will dispose of this suit.

First, validity. The use of phytase to break down phytic acid—the basic chemical reaction claimed by the patents-in-suit—was known in the prior art, and defendants have shown that the patents-in-suit merely claim a new use for known techniques and materials. Each element of the asserted claims in both of the patents-in-suit was disclosed, either expressly or inherently, in a single prior art reference. Thus, those claims are invalid as anticipated under 35 U.S.C. § 102. The court will grant summary judgment to defendants on this issue.

Second, inequitable conduct. Defendants have not adduced evidence sufficient to sustain their burden to show either the materiality of the withheld information or the deceptive intent of the patentee. The court will grant summary judgment to plaintiffs on this issue.

BACKGROUND

The following facts are undisputed, except where noted.

A. The parties

U.S. Water Services is a water treatment company, headquartered in St. Michael, Minnesota. Roy Johnson is the company's Chief Innovation Officer and a co-inventor of the patents-in-suit. Mr. Johnson assigned his rights in these patents to U.S. Water. In this opinion, the court will refer to plaintiffs collectively as U.S. Water, except when separately identifying them. Novozymes A/S is a global biotechnology company, based in Bagsvaerd, Denmark. Novozymes North America is a subsidiary of Novozymes A/S, incorporated in New York and based in Franklinton, North Carolina. In this opinion, the court will refer to defendants as Novozymes.

B. Technology at issue

The technology at issue relates to an enzyme, phytase, which is useful in fuel ethanol production. The essential aspects of ethanol production are undisputed, although the parties disagree on some details that are not material to this decision.

Ethanol is commonly produced by using yeast to ferment starchy grains, such as corn, wheat, or sorghum. In "dry grind" ethanol plants, these grains are milled to produce a meal, which is then mixed with water in a slurry tank to form a mash.[1] Enzymes are added to the mash, which then travels to liquefaction tanks to sit for a few hours, and to allow the enzymes to begin breaking down the starches into sugars. Following liquefaction, the mash moves into fermentation tanks, where yeast digests the sugars, producing alcohol and carbon dioxide. In

[1] The parties agree that fuel ethanol can also be produced using a "wet milling" process, which is similar in overall concept. Dkt. 267, at 17-18. According to the parties, there are no corn wet milling facilities that are dedicated exclusively to the production of fuel ethanol. *Id.* at 18.

some ethanol plants, yeast is added directly into the fermentation tanks; in others, the yeast is added through a propagator tank, which hydrates the yeast before transferring it to the fermentation tank.

When fermentation is complete, the resulting product—referred to as beer—is transferred to a distillation column, called a beer column. Some ethanol plants use a holding tank, called a beer well, to store the beer before it enters the beer column, but others transfer the beer directly to the beer column. In plants that use beer wells, the beer passes through a beer mass heat exchanger before entering the beer column. Regardless of whether a plant uses a beer well, distillation begins once the beer reaches the beer column. Beer enters near the top of the beer column, and then cascades down over a series of horizontally stacked trays. Meanwhile, steam is added to the bottom of the column and works its way upward, capturing vaporized ethanol from the beer as it does so. The ethanol-containing vapor exits the beer column at the top to be concentrated using molecular sieves. The product remaining in the beer column is called whole stillage. Whole stillage can be further broken down into several products, including backset and thin stillage. Ethanol plants recycle these products for use as process water for the next batch of mash. Other byproducts can be used in animal feed.

A common problem in fuel ethanol production is "fouling," wherein deposits form on the machinery that contacts the mash, fermenting broth, or ethanol processing fluid that has poorly converted starch. These deposits impede heat transfer and fluid flow, decreasing the efficiency of the affected machinery. Fouling is a significant problem: according to a Novozymes presentation produced during discovery, fouling results in downtime that costs the ethanol industry in the United States $71 million per year.[2]

[2] Novozymes objects to this evidence on the grounds that U.S. Water has not identified a witness with personal knowledge of the presentation through whom the evidence could be

Traditional solutions to fouling include physically removing the deposits using a high pressure water jet and cleaning machinery with sulfuric acid or caustic cleaners. Alternatively, ethanol plants can increase the solubility of deposit-forming material, allowing the material to dissolve in the beer rather than form deposits on the machinery. A common way of doing this is adding sulfuric acid at one or more points during the production process. Sulfuric acid reduces the pH of the beer which, in turn, increases the solubility of deposit-forming material. Physically cleaning the machinery and using sulfuric acid to lower pH are expensive: U.S. Water estimates that these measures may cost individual plants hundreds of thousands of dollars per year.[3]

This case is about a different type of solution to the problem of fouling, which uses the enzyme phytase. The processing fluid used during ethanol production contains phytic acid, and its metallic salts, which are a prime cause of fouling. Phytic acid and its metallic salts precipitate out of the ethanol processing fluid, forming deposits as the fluid travels through the machinery. Phytase helps prevent these deposits from forming by chemically breaking down the phytic acid. Once broken down, phytic acid byproducts are more readily dissolved in the processing fluid. Using phytase to control deposits is cheaper than using sulfuric acid or physically cleaning fouled machinery.

C. The patents in suit

There are two patents in suit: U.S. Patent No. 8,415,137, "Preventing Phytate Salt Deposition in Polar Solvent Systems," to Roy Johnson and Paul R. Young; and U.S. Patent No.

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introduced at trial. Dkt. 234, at 12. Whatever the merit of the evidentiary objection, Novozymes does not contend that the information contained in its presentation is inaccurate.

[3] Novozymes quibbles that the actual cost depends on the size and set-up of the plant, the frequency of cleanings, market conditions, and other variables. Dkt. 234, at 14. This dispute is immaterial: the parties generally agree that physical cleaning and sulfuric acid are expensive responses to fouling.

8,609,399, "Reducing Insoluble Deposit Formation in Ethanol Production," also to Johnson and Young. Both patents claim priority to U.S. Patent Application 11/873,630, which issued as U.S. Patent No. 8,039,244. The parent '630 application was filed on October 17, 2007, which is thus the priority date of the patents-in-suit.

The three patents share a common specification which teaches the value of using phytase to reduce deposits in ethanol production equipment and describes methods of doing so. The '244 patent claimed a method of reducing deposits by adding phytase to thin stillage or backset, which is to say, adding phytase *after* fermentation.[4] The '137 patent and the '399 patent contain broader claims. Both patents claim adding phytase to "an ethanol processing fluid," thus apparently extending the scope of the invention by claiming methods in which phytase is added before or during fermentation. The asserted claims of the '137 patent and the '399 patent also include limitations on the conditions for adding the enzyme, identifying ranges for dosage, temperature, and pH level. The asserted claims include limitations that require breaking down phytic acid and reducing deposits substantially without adding an acidic compound.

The court will defer a more detailed discussion of the asserted claims to the validity analysis below.

D. Prior art

There are two prior art references pertinent to this opinion. The first is a World Intellectual Property Organization application, No. WO 01/62947 for "Fermentation with a Phytase," to Chris Veit and others, published on August 30, 2001. The court will refer to this published application as "Veit." Veit has an extensive prosecution history, which is generally immaterial to the court's decision because Veit's priority over the patents-in-suit is clear. According to Veit's specification, the "invention relates to a process of fermenting phytic acid-containing materials; a process of alcohol and other fermented compounds production, in particular ethanol production; the use of phytase activity for saccharification and/or fermentation; and a composition suitable for ethanol production." Veit, 1:4-9.

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[4] The scope of the claims of the '244 patent was previously litigated. *U.S. Water Servs., Inc. v. ChemTreat, Inc.*, No. 11-cv-0895, 2013 WL 173736, at *1 (D. Minn. Jan. 16, 2013), *appeal transferred*, 570 F. App'x 924 (Fed. Cir. 2014), *and aff'd*, No. 14-3057, 2015 WL 4491398 (8th Cir. July 24, 2015). For purposes of this case it is thus established that the '244 patent claims only the addition of phytase after fermentation. Facts related to this litigation are a central part of Novozymes's allegation of inequitable conduct, addressed below.

The second pertinent prior art reference is United States Patent No. 5,756,714, for "Method for Liquefying Starch," to Richard L. Antrim and others. The court will refer to this patent as "Antrim." Antrim issued in 1998, claiming priority to an application filed March 27, 1995. As with Veit, Antrim's priority over the patents-in-suit is clear and undisputed. Antrim's claims describe a method for treating "starch prior to or simultaneously with liquefying said starch to inactivate and/or remove an enzyme inhibiting composition present in said starch to form treated starch." Antrim, 20:10-13.

E. Novozymes's phytase products

Novozymes's alleged infringement is not material to the validity of the patents-in-suit. But to provide a reasonably complete background to the case, the court provides a brief overview of the activities that U.S. Water alleges to be infringing. U.S. Water has developed a phytase-based product, which it has marketed under the brand name pHytOUT. Since 2009, U.S. Water has offered pHytOUT to its ethanol plant customers as part of a method for deposit control.

Novozymes started offering a phytase-based fouling-reduction product for ethanol processing in 2011, which it branded as Phytaflow beginning in 2013. U.S. Water alleges that Novozymes's customers directly infringe the patents-in-suit by practicing the claimed methods of deposit reduction, and that Novozymes indirectly infringes these patents by inducing its customers to directly infringe, or by contributing to their direct infringement by supplying them with Phytaflow. U.S. Water has identified eight ethanol plants—none of which are parties to this action—that it contends use Novozymes's Phytaflow to reduce fouling. Some of these plants are former U.S. Water customers, and U.S. Water has adduced evidence that these plants switched to Novozymes's product because it was less expensive.

ANALYSIS

A. Jurisdiction and basic legal principles

The case arises under the patent laws of the United States, and the court has subject matter jurisdiction under 28 U.S.C § 1338. The court looks to Federal Circuit precedent when considering substantive issues of patent law, and to the regional circuit for procedural law. *Aero Products Int'l, Inc. v. Intex Recreation Corp.*, 466 F.3d 1000, 1016 (Fed. Cir. 2006). But as in standard civil cases, summary judgment in a patent case is appropriate if "the movant shows that there is no genuine dispute as to any material fact and the movant is entitled to judgment as a matter of law." Fed. R. Civ. P. 56(a). The court views all facts in the light most favorable to the non-moving party. *Anderson v. Liberty Lobby, Inc.*, 477 U.S. 242, 255 (1986). "Only disputes over facts that might affect the outcome of the suit under the governing law will properly preclude the entry of summary judgment." *Id.* at 248. Where there are cross-motions for summary judgment on issues of infringement and invalidity, as is true in this case, "the court views the record evidence through the prism of the evidentiary standard of proof that would pertain at a trial on the merits." *SRAM Corp. v. AD-II Eng'g, Inc.*, 465 F.3d 1351, 1357 (Fed. Cir. 2006) (citations omitted).

U.S. Water's patents are presumed valid under 35 U.S.C. § 282(a). That presumption is overcome on anticipation grounds only if Novozymes adduces clear and convincing evidence that at least one piece of prior art anticipates the patents-in-suit. Anticipation is a question of fact, which may be decided on summary judgment "if no genuine issue of material fact exists [and] if no reasonable jury could find that the patent is not anticipated." *OSRAM Sylvania, Inc. v. Am. Induction Technologies, Inc.*, 701 F.3d 698, 704 (Fed. Cir. 2012) (internal citations and quotation marks omitted).

"Under 35 U.S.C. § 102[,] a claim is anticipated if each and every limitation is found either expressly or inherently in a single prior art reference." *King Pharm., Inc. v. Eon Labs, Inc.*, 616 F.3d 1267, 1274 (Fed. Cir. 2010) (internal citations and quotation marks omitted). Determining whether claims are anticipated involves two steps: (1) construing the claims of the patents-in-suit; and (2) comparing the construed claims to the prior art. *Enzo Biochem, Inc. v. Applera Corp.*, 599 F.3d 1325, 1332 (Fed. Cir. 2010). Although a prior reference must arrange or combine the elements of the claimed invention in the same way as the claim, the reference need not disclose the elements in precisely the same language. *Whitserve, LLC v. Computer Packages, Inc.*, 694 F.3d 10, 21 (Fed. Cir. 2012). This case primarily involves the principle of inherent disclosure, according to which "a prior art reference may anticipate without disclosing a feature of the claimed invention if that missing characteristic is necessarily present, or inherent, in the single anticipating reference." *Schering Corp. v. Geneva Pharm.*, 339 F.3d 1373, 1377 (Fed. Cir. 2003) (citations omitted).

B. Anticipation by Veit and Antrim

1. The asserted claims

U.S. Water asserts 29 claims in this case: from the '137 patent, one independent and three dependent claims; from the '399 patent, four independent and 21 dependent claims. Dkt. 204, at 102.[5] The validity of each of these claims must be separately evaluated, because the

[5] On June 9, 2015, the parties stipulated to dismiss U.S. Water's claims of infringement relating to four of the originally asserted claims in the '399 patent. Dkt. 307.

invalidity of an independent claim does not, by itself, entail the invalidity of those claims that depend from it. 35 U.S.C. § 282(a).

Although each claim must in some sense be separately considered, the claims share common elements. Novozymes proposes (and U.S. Water does not disagree) that the asserted claims, collectively, contain various combinations of seven limitations. Those limitations are:

(1) adding phytase to a fuel ethanol processing fluid containing phytic acid or phytic acid salts;

(2) at a dosage of 10 ppm less or 50 U/L or less;

(3) at a temperature between 20℃ and 80℃;

(4) at a pH of 4.5 or higher in the beer column; and

(5) without the addition of an acidic compound, an oxidizer, and oxidizing agent, or ultraviolet light;

(6) breaking down phytic acid;

(7) thereby reducing the formation of insoluble deposits.

Dkt. 200, at 66.

Claim 1 of the ’399 patent is a representative independent claim:

> 1. A method of reducing formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during the production of a quantity of ethanol, wherein the production of a quantity of ethanol comprises a fermentation step and wherein the fuel ethanol processing plant comprises a piece of heat transfer equipment, the method comprising:
>
> providing an additive in an ethanol processing fluid in the plant, wherein the ethanol processing fluid comprises an amount of phytic acid and/or salts of phytic acid, and wherein the additive comprises phytase,
>
> wherein the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid by breaking down the phytic acid and/or phytic acid salts,
>
> wherein providing the additive comprising phytase in the ethanol processing fluid causes a reduction of the formation

> of insoluble deposits of phytic acid and/or salts of phytic acid in a piece of heat transfer equipment in the plant, and
>
> wherein the reduction in the formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during production of the quantity of ethanol is accomplished substantially without the addition of an acidic compound that can break down organic phosphates and phosphonates into soluble inorganic phosphates in the presence of an oxidizer, oxidizing agent, or ultraviolet light.

’399 patent, at 12:30-57.

Claims 5 and 6 are representative dependent claims:

> 5. The method of claim 1, wherein the plant further comprises a beer column and wherein providing the ethanol processing fluid comprising phytase also causes the amount of insoluble phytic acid and/or salts of phytic acid deposited in the beer column to be reduced.
>
> 6. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 500 ppm or less.

Id. at 13:21-29.

2. Claim construction

The parties do not identify anticipation-specific claims construction issues, although they have contested four claim terms in the context of infringement. Two of these contested terms are arguably material to the question of anticipation, and thus the court will construe those terms.

A “bedrock principle” of patent law is that “the claims of a patent define the invention to which the patentee is entitled the right to exclude.” *Phillips v. AWH Corp.*, 415 F.3d 1303, 1312 (Fed. Cir. 2005) (en banc) (citations and quotation marks omitted). The patent’s intrinsic evidence is the “primary basis for construing” a claim and “the best source for understanding a technical term." *Id.* at 1315 (citations and quotation marks omitted). Intrinsic evidence includes the patent and its prosecution history, related patents and their prosecution histories, and the prior art that is cited or incorporated by reference in the patent-in-suit and prosecution history. *Id.* at 1315-17. Of these sources, "the specification is always highly relevant to the claim construction analysis. Usually, it is dispositive; it is the single best guide to the meaning of a disputed term." *Id.* at 1315 (internal citations and quotation marks omitted).

The court may also consider extrinsic evidence, which refers to all other types of evidence, including inventor testimony, expert testimony, documentary evidence of how the patentee and alleged infringer have used the claim terms, dictionaries, treatises, and other similar sources. *Id.* at 1317-18. Extrinsic evidence may assist the court in understanding the underlying technology, the meaning of terms to one skilled in the art, and how the invention works. *Id.* at 1317-19. However, extrinsic evidence is less reliable and less useful in claim construction than the patent and its prosecution history. *Id.* Intrinsic evidence trumps any extrinsic evidence that would contradict it. *Id.* at 1314.

a. "Wherein the pH of the ethanol processing fluid in the beer column is 4.5 or higher during production of the quantity of ethanol"

The first disputed term relates to the pH range that the patents-in-suit claim. According to the parties, proper construction of this term turns on the word "during." Novozymes contends that it means "at all times during," so that the claim requires the pH in the beer column to always be above 4.5. U.S. Water proposes a less rigid construction, contending that the term "during" means "at some point in." The specification confirms that the claimed invention covers more than a method for combating deposits that occur *exclusively* at pH levels above 4.5, although this was a principal focus of the invention. And the extrinsic evidence supports a less rigid construction as well. A person of ordinary skill in the art would understand the practical impossibility of maintaining a consistent pH level in the beer column, and would not read the term as requiring that the pH always stay above 4.5 for the invention to work.

The word "during" appears throughout the specification and the asserted claims, sometimes referring to a continuous event or condition, and sometimes referring to a single occurrence. For example, the specification teaches that "[a] small amount of phytic acid is naturally broken down into soluble byproducts . . . during fermentation," '399 patent, 4:24-26, which presumably refers to an ongoing reaction. But the specification also describes introducing mash "into [a] fermentation tank during ethanol processing," *id.* at 2:11-15, and it provides an aspect of the invention wherein phytase is introduced "into thin stillage or backset-containing equipment during ethanol processing," *id.* at 6:21-24. These are both events that are started and completed in ethanol processing, and that therefore do not occur "at all times" throughout the process.

The substance of the common specification helps resolve any ambiguity. According to the patents-in-suit, fouling occurs at a range of pH levels. "[D]eposits tend to be most severe or tenacious on hot surfaces, and where the pH of the processing liquid is highest (about 4.5), but deposits may also form at lower pH values and on cooler surfaces." '399 patent, 1:38-43. The specification includes a table disclosing that when phytic acid is in a solution containing ethanol (as it would be in a beer column), precipitate becomes visible at a pH of 4.35 and a temperature of 40°C. *Id.* at 5:7-19. Thus, the specification acknowledges that the pH does not need to be above 4.5 for the claimed invention to successfully reduce deposits. Indeed, even the targeted "tenacious" deposits occur at *about* 4.5, which would include lower values. Given the breadth of the specification, as well as its disclosure that phytase can reduce deposits at lower pH levels, it would be inconsistent to construe this term in a way that would not cover a process in which the pH level in the beer column dipped below 4.5 at some point. Moreover, the specification does not disclose a method for maintaining the pH above 4.5 once the ethanol processing fluid enters the beer column, an instruction that practitioners would expect to see if the inventors truly meant to claim an invention that required a pH above 4.5 at all times.

Novozymes's intrinsic evidence is meager in comparison. While prosecuting the '137 patent, the inventors at one point informed the PTO that they wished to "restrict" pH in the beer column because that is one of the sites at which deposits are reduced. Dkt. 208-4, at 49. But even in this statement, the inventors did not indicate that the pH must stay above 4.5 *at all times*. Considered along with the specification, the inventors' response merely confirms that they wanted to emphasize how phytase could prevent fouling that occurs at higher pH levels.

The extrinsic evidence supports construing the disputed term to mean "at some point in." U.S. Water has provided comprehensive and generally undisputed expert evidence regarding how a person of ordinary skill in the art would read the term. Courts must construe claims to give them "the meaning that the term would have to a person of ordinary skill in the art in question at the time of the invention." *Phillips*, 415 F.3d at 1313. The parties agree that a person of ordinary skill in the art would have an understanding of the operation of fuel ethanol processing plants. Dkt. 267, at 259. For example, someone skilled in the art would know that some plants monitor pH levels in the beer column and set target pH levels to accomplish deposit reduction, while other plants do not monitor pH levels. But even the plants that monitor pH levels do not always have precise control systems, and so the actual pH of the beer frequently fluctuates above and below any target that the plant sets. And finally, adjusting the pH takes time; it does not happen instantaneously. Given this inherent variability, U.S. Water's expert, Mr. Simms, opines that a person of ordinary skill in the art would not construe "during" to mean "at all times." Dkt. 174, ¶ 70. Instead, Mr. Simms suggests that a person of ordinary skill in the art would understand "during" to require that the pH in the beer column be above 4.5

"for a reasonable period of time." *Id.* ¶ 72. U.S. Water explains that the expert's construction, although using different words, is substantively the same as its own proposed construction because neither interprets "during" in a way that would allow U.S. Water to claim de minimus infringement. Dkt. 265, at 78. U.S. Water's construction is based on the practicalities of ethanol production that a person of ordinary skill in the art would incorporate into his or her reading of the disputed term. Thus, the extrinsic evidence supports construing the term to mean "at some point."

The court construes the claim "wherein the pH of the ethanol processing fluid in the beer column is 4.5 or higher during production of the quantity of ethanol" to mean that the pH must be 4.5 or higher at some point during production.

b. "Wherein the reduction . . . is accomplished substantially without the addition of an acidic compound"

The second claim that the court must construe concerns the extent to which plants must be able to reduce deposits with phytase, as opposed to with an acidic compound and an oxidizing agent or with an acidic compound and ultraviolet light. Novozymes contends that this term means that plants practicing U.S. Water's invention must accomplish deposit reduction without using an acidic compound at any time in the production process. U.S. Water contends that the plain meaning is sufficient and no further construction is necessary. In the alternative, U.S. Water proposes that the court construe the term to mean that deposit reduction is accomplished substantially by phytase and not by the addition of an acid compound.

The intrinsic evidence does not support Novozymes's construction. The specification discloses three ways to reduce deposits by adding an "agent" to ethanol processing fluid. '399 patent, 5:21-37. In the first two methods, the agent is an acidic compound (*e.g.*, sulfuric acid); one method combines the acid with an oxidizer or oxidizing agent, the other method combines the acid with ultraviolet light. In the third method, the agent is an enzyme capable of degrading organic phosphates (*e.g.*, phytase). During the prosecution of the '137 patent, the applicants included a claim that limited the invention to cover reductions "accomplished substantially without the addition of *an acid*." Dkt. 280, at 102 (emphasis added). The applicants relied on the above-cited portion of the specification to support the limitation. *Id.* But the PTO initially rejected the limitation, concluding that it lacked support in the specification. *Id.* In response, the applicants re-emphasized the above-cited language, and they informed the PTO that they meant to claim a method for using the phytase-based agent to the exclusion of the two agents. *Id.* at 103-04.

Claim 1 of the '399 patent illustrates the import of the applicants' statements to the PTO. The preamble claims "[a] method of reducing formation of insoluble deposits." '399 patent, 12:30. The preamble is followed by four clauses that delineate the method, one of which contains the disputed term. Clause one claims adding phytase. Clause two claims that the phytase will reduce the amount of phytic acid. Clause three claims that adding phytase "causes a reduction of the formation of insoluble deposits." *Id.* at 12:45-46. Finally, clause four claims that the reduction (*i.e.*, the one claimed in clause three) "is accomplished substantially without the addition of an acidic compound that can break down organic phosphates and phosphonates into soluble inorganic phosphates in the presence of an oxidizer, oxidizing agent, or ultraviolet light." *Id.* at 52-57. Read in its entirety, and against the backdrop of the specification, this claim aligns with what the applicants told the PTO. The claim states that the reduction in deposits will be accomplished by phytase, rather than by acid in combination with an oxidizer or by acid in combination with ultraviolet light. Construing the disputed term as Novozymes proposes ignores this prosecution history and does not conform to the claim language.

The court will construe the term "wherein the reduction . . . is accomplished substantially without the addition of an acidic compound" to mean that deposit reduction is accomplished substantially by phytase and not by the addition of an acid compound with an oxidizer or with ultraviolet light. But the term does not preclude all use of acidic compounds in ethanol processing.

3. Comparison of the claims with Veit and Antrim

Novozymes contends that Veit and Antrim each expressly disclose the first six of the claim elements listed above and that both references inherently disclose the seventh element, which concerns the purpose of the invention. U.S. Water contends that the references no not disclose elements two, four, and seven. *See generally* Dkt. 226, at 95-119. The evidence of record shows beyond genuine dispute that the expressly disclosed elements, one through six, are disclosed in Veit and Antrim. Thus, the critical issue in this case is the inherent disclosure of adding phytase for the purpose of reducing deposits.

The fundamental flaw in U.S. Water's argument is that "[n]ewly discovered results of known processes directed to the same purpose are not patentable because such results are inherent." *Bristol-Myers Squibb Co. v. Ben Venue Labs., Inc.*, 246 F.3d 1368, 1376 (Fed. Cir. 2001) (citations omitted). Here, the evidence of record establishes that, at best, U.S. Water discovered a new use for the method expressly disclosed in Veit and in Antrim. U.S. Water therefore cannot genuinely dispute that its asserted claims are invalid under 35 U.S.C. § 102, and the court will enter summary judgment in Novozymes's favor.

c. Express disclosure

Veit and Antrim expressly disclose the first six elements of the above-identified list. U.S. Water challenges only whether Veit discloses the dosage range and the pH of 4.5 or higher in the beer column, *see* Dkt. 226, at 98-108, and for Antrim, U.S. Water challenges only whether the reference expressly discloses a pH of 4.5 or higher in the beer column, *id.* at 108-13. Before addressing each element, the court notes that three of these claim elements concern ranges for the conditions under which phytase is added to ethanol processing fluid. "[W]hen a patent claims a chemical composition in terms of ranges of elements, any single prior art reference that falls within each of the ranges anticipates the claim." *Atlas Powder Co. v. Ireco, Inc.*, 190 F.3d 1342, 1346 (Fed. Cir. 1999). Thus, it is enough for Veit or Antrim to expressly disclose values or ranges of values that fall within the ranges contained in the patents-in-suit. The ranges do not need to precisely overlap, nor does the prior art's range need to fit entirely within the range claimed by the patents-in-suit. *Perricone v. Medicis Pharm. Corp.*, 432 F.3d 1368, 1377 (Fed. Cir. 2005) (holding that a range of amounts of acid that did not exactly correspond to prior art was nonetheless anticipated by the prior art because the prior art range entirely encompassed the range of the patent-in-suit and did not significantly deviate from it).

1. Elements one, three, five, and six

The court will succinctly address elements one, three, five, and six because U.S. Water does not challenge Novozymes's contention that Veit and Antrim each expressly disclose these elements.

The first element of the asserted claims is adding phytase to a fuel ethanol processing fluid containing phytic acid or phytic acid salts. Both Veit and Antrim expressly disclose this element. Veit, 19:2-4; Antrim, 20:8-19. Although U.S. Water identifies semantic discrepancies, the language of the prior art precludes any genuine dispute on this point. Veit discloses "a process including a fermentation step, wherein phytic acid-containing material is fermented in the presence of a phytase." Veit, 19:2-4. Veit also discusses ethanol production. *Id.* at 2:15-29. Antrim teaches adding phytase while converting "grain starch to downstream products, such as dextrose, fructose[,] and alcohol." Antrim, 1:10-16. rim primarily teaches using phytase to improve liquefaction, *id.* at 4:6-8, although U.S. Water does not suggest that this emphasis render's Antrim inapplicable to ethanol production. Instead, U.S. Water merely notes that Antrim is directed to a process that occurs well before the fermentation and distillation steps of ethanol production. Dkt. 226, at 109. But Antrim does not teach removing phytase after liquefaction is complete, and U.S. Water does not otherwise explain how adding phytase earlier during ethanol production will prevent the enzyme from operating in accordance with the claimed invention later in the process.

The third element limits the range of temperatures within which phytase is added to ethanol processing fluid. There is no dispute that Veit and Antrim each disclose the temperature range claimed by the patents-in-suit. Veit states that "[i]n a preferred embodiment[,] the phytase has a temperature optimum in the range from 25-70℃, preferably 28-50℃, especially 30-40℃." Veit, 8:4-6. Indeed, Veit claims a method wherein the optimal range is "20-70℃, in particular above 50℃." *Id.* at 19:8-9. Antrim states that "[f]or microbial phytase, a suitable temperature will generally be between about 20℃ and about 60℃, and preferably between about 30℃ and about 40℃." Antrim, 7:60-62. Both references disclose sets of ranges that overlap completely with the 20-80℃ range that the patents-in-suit claim, and both references therefore expressly disclose element three. *See Atlas Powder Co.*, 190 F.3d at 1346.

Applying U.S. Water's proposed claim construction, the fifth element of the asserted claims requires adding phytase without also adding: (1) an acidic compound and an oxidizer; or (2) an acidic compound in the presence of ultraviolet light. It is undisputed that both Veit and Antrim disclose adding phytase. Veit's claimed invention does not require adding an acidic compound, let alone adding an acidic compound with an oxidizer or in the presence of ultraviolet light. U.S. Water does not contend that an express statement exclusion is required, and so the court is satisfied that Veit discloses this element. Likewise, Antrim does not require adding an acidic compound along with phytase. Indeed, the reference teaches that by adding phytase, "the need to undesirably adjust the pH level . . . is eliminated." Antrim, 7:43-45. And if Antrim discloses not adding acid, then it necessarily discloses not adding acid with an oxidizer or in the presence of ultraviolet light. Both references expressly disclose the non-addition of an acidic compound.

The sixth element describes what phytase does, and both Veit and Antrim expressly disclose that the enzyme breaks down phytic acid. Veit states that the phytase is "capable of effecting the liberation of inorganic phosphate from phytic acid (myo-inositol hexakisphosphate) or from any salt thereof (phtyates)." Veit, 7:31-34. Antrim describes "a phytate degrading enzyme" that "catalyze[s] the conversion of phytate to inositol and inorganic phosphate." Antrim, 6:41-52. As U.S. Water observes, the purpose for which Veit and Antrim direct using phytase is not deposit reduction. But element six is not concerned with purpose; what matters is whether the references explicitly describe using phytase to break down phytic acid. Here, both Veit and Antrim satisfy this requirement.

2. Element two: "at a dosage of 10 ppm less or 50 U/L or less"

The first challenged element is whether Veit or Antrim expressly disclose the claimed dosage. The patents-in-suit claim a dosage for phytase of less than 10 ppm or 50 U/L. Veit states that "[t]he dosage of the phytase may be in the range 5.000-250.000 FYT/g DS, particularly 10.000-100.000 FYT/g DS. A preferred suitable dosage of the phytase is in the range from 0.005-25 FYT/g DS, preferably the 0.01-10 FYT/g, such as 0.1-1 FYT/g DS." Veit, 8:11-15. Antrim describes a dosage of "from about 0.1 to 100 units of phytase (phytase unit) per gram of starch. More preferably, the concentration of phytate degrading enzyme is from about 1 to about 25 units of phytase per gram of starch." Antrim, 7:6-11. Because the patents-in-suit use different units to measure dosage, Novozymes's expert, Dr. Kohl, translated these units to compare them to the units used in the patents-in-suit. His translation is expressed in the following table.

137 Patent Claims	399 Patent Claims	Asserted Patents (ppm phytase)	Asserted Patents (U/L)	Veit app. (FYT/g DS)	Antrim patent (units phytase /g starch)
12	6, 17, 25	500	550,110	1667	2,381
		160	176,000	476	680
	7, 18, 28	100	109,890	333	476
		75	82,500	250	357
		30	33,000	100	143
		21	23,100	70	100
	8, 19, 23, 29	20	22,000	67	95
	9, 20, 30	10	11,000	33	48
		3	3,300	10	14
13	10, 21, 25	2.3	2,500	7.6	11
		1.6	1,779	5.4	7.7
	11, 22, 31	0.5	500	1.52	2.2
		0.16	176	0.53	0.76
	12, 32	0.09	100	0.30	0.43
	13, 24, 33	0.05	50	0.152	0.22
		0.021	23	0.07	0.10
		0.002	1.8	0.00539	0.008
		0.0015	1.65	0.005	0.007

As Dr. Kohl's table shows, both Veit and Antrim disclose dosage ranges that overlap with the range claimed by the patents-in-suit.

U.S. Water attempts to dispute that the dosage concentrations of the patents-in-suit are disclosed by Veit and Antrim by citing inaccuracies in Novozymes's expert's conversion calculations.[6] Dkt. 235, at 227-30. But U.S. Water's challenges to some of the details of the

[6] Part of U.S. Water's contention is based on its speculation that Veit used the European numbering convention in which a period is used as the thousands separator and a comma is used as a decimal point. Dkt. 226, at 108 and Dkt. 235, at 211. This does not raise a genuine dispute because there is nothing in Veit that suggests it used the European convention. Veit uses periods for pH values, confirming the traditional English numbering convention. Veit's

conversion are immaterial because U.S. Water concedes that the prior art discloses dosages that fall within the ranges claimed by the patents-in-suit. *See* Dkt. 200, at 88; and Dkt. 279, at 213-15 (U.S. Water's response to Novozymes's proposed fact 300).

3. Element 4: "at a pH of 4.5 or higher in the beer column"

The patents-in-suit claim a pH range of 4.5 and higher in the beer column. Following U.S. Water's own construction of this term, the limitation requires only that the pH in the beer column exceed 4.5 at some point during ethanol production. Veit discloses a process wherein fermentation is completed in the presence of phytase and when "the pH is from pH 3-6, preferably around pH 4-5." Veit, 4:7-9, 19:1-4. Antrim discloses liquefying starch with a pH "between 4 and about 6, . . . [p]referably between about 4.5 and about 5.2." Antrim, 7:39-53. As with the temperature and dosage ranges, this overlap is enough to expressly anticipate the asserted claims. *Perricone*, 432 F.3d at 1377; *Atlas Powder Co.*, 190 F.3d at 1346. U.S. Water therefore cannot genuinely dispute that the prior art discloses using phytase when the pH is 4.5 or higher.

But this element has a second component. The claimed pH must occur in the beer column. U.S. Water suggests that a person of ordinary skill in the art would change the pH after liquefaction (as described in Antrim) and after fermentation (as in Veit). Thus, U.S. Water contends that Veit and Antrim do not necessarily disclose a pH of 4.5 or higher at some point in the beer column. This argument is not persuasive for two reasons. First, U.S. Water improperly relies on an untimely declaration of one of its experts. According to Mr. Simms, "it was well

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published U.S. application, US 2007/0155001 A, Dkt. 243-8, is immaterial. And the ranges cited in that document (5,000-250,000 FYT/g DS and 10,000-100,000 FYT/g DS) are probably mistaken, for reasons explained by Novozymes. Dkt. 279, at 213-15 (reply to proposed fact 300). The rest of U.S. Water's argument is that the calculations are based on assumptions that are not accurate. *Id.* at 212-15, 221-23. Despite this quibbling, U.S. Water does not dispute the larger point: Veit and Antrim's dosages overlap with the patents-in-suit.

known that fuel ethanol plants prior to 2007 [when U.S. Water applied for the '244 patent] routinely added sulfuric acid to their beer feed in order to reduce the pH of the beer going into their beer mash heat exchangers and beer columns to well below 4.5." Dkt. 230, ¶ 16. Thus, a pH of 4.5 during fermentation may not necessarily mean a pH of 4.5 in the beer column during distillation. Indeed, Mr. Simms opines that "one of ordinary skill in the art would not read any prior art [including Veit and Antrim] to describe the pH of the ethanol processing fluid in a beer column to be 4.5 or higher." *Id.* Because the court must consider whether one skilled in the art would reasonably understand the prior art to disclose every element of the asserted claims, *see Dayco Prods., Inc. v. Total Containment, Inc.*, 329 F.3d 1358, 1368-69 (Fed. Cir. 2003) (citing *In re Baxter Travenol Labs.*, 952 F.2d 388, 390 (Fed. Cir. 1991)), U.S. Water contends that its expert's declaration is sufficient to defeat Novozymes's anticipation defense.

Novozymes has moved to strike Mr. Simms's declaration as untimely. Dkt. 273. Opening expert disclosures in this case were due November 18, 2014, with rebuttal reports due December 18, 2014. Dkt. 275-2 (amending the expert disclosure dates, pursuant to the court's Preliminary Pretrial Conference Order). In his opening report on invalidity, Novozymes's expert explained that Veit and Antrim disclose a pH of 4.5 or higher in the beer column because "[f]ermentation pHs between 5.5 and 6.0 will result in post-fermentation fluid in the beer column with a pH above 4.5." Dkt. 177, ¶ 145. Mr. Simms's rebuttal validity report did not respond to these opinions, instead emphasizing that the prior art references did not expressly disclose the same dosage range and did not inherently disclose using phytase for the purpose of reducing deposits. *See* Dkt. 174, ¶¶ 356-75, 389-401. Mr. Simms's supplemental declaration therefore improperly injects a brand new expert opinion regarding validity.[7]

[7] U.S. Water effectively concedes that Mr. Simms's supplemental declaration ventures into different territory than his initial report covered. As U.S. Water acknowledges, the "implicit"

The Preliminary Pretrial Conference Order explicitly warned the parties that "[s]upplmentation [of expert reports] pursuant to Rule 26(e) is limited to matters raised in an expert's first report. . . . Failure to comply with these deadlines and procedures could result in the court striking the testimony of a party's experts pursuant to [Federal Rule of Civil Procedure] 37." Dkt. 37, at 3. Under Rule 37(c)(1), a party may not use information that it should have, but failed to, disclose under Rule 26(a), unless such failure was substantially justified or harmless. Here, U.S. Water's non-disclosure of this aspect of Mr. Simms's validity opinion is neither. As justification, U.S. Water offers that "[t]he preparation of expert reports was an enormous undertaking," and that it should "come as no surprise" that an issue addressed in an initial expert report might require further elaboration. Dkt. 288, at 8. This is a poor excuse. Novozymes has made no secret of its contention that Veit and Antrim are anticipating prior art, and the opening invalidity report of its expert walked through each limitation of the asserted claims and explained how Veit and Antrim expressly disclose those limitations. Contrary to U.S. Water's assertion, the additional information presented in the Simms declaration is not "minor;" it goes directly to the issue of anticipation. The fact that this case took considerable effort to prepare and present simply does not justify allowing U.S. Water to present an expert opinion on validity well beyond the deadline for doing so.

Nor is U.S. Water's failure to disclose this aspect of Mr. Simms's opinion harmless. Novozymes filed its opening summary judgment brief on January 16, 2015. The Simms declaration was filed on February 6, 2015. U.S. Water is therefore incorrect that Novozymes suffered no prejudice or that it had time to cure any prejudice that it did suffer. Novozymes was

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opinion (in his initial report) that Mr. Simms's declaration purports to supplement is a statement regarding claims construction. Dkt. 288, at 6. Some 100 pages later, when Mr. Simms opines on the issue of validity in view of Veit, he does not refer to his earlier statement about the common practice of lowering the pH of beer before it enters the beer column.

entitled to rely on Mr. Simms's opinions while formulating its summary judgment arguments, and having to scramble to address the issue in a reply brief—due just 17 days after the Simms declaration was filed—is hardly adequate time to reframe its theory of invalidity. For these reasons, the court will grant Novozymes's motion to strike paragraphs four to six and 16 of the Simms declaration.[8]

A second reason to reject U.S. Water's argument is that even Mr. Simms's untimely opinion does not suggest that adding phytase as disclosed in Veit or Antrim will *never* result in a pH above 4.5 in the beer column. Applying U.S. Water's own construction, all that this term requires is that the pH be above 4.5 *at some point* during ethanol production. The common specification for the patents-in-suit does not disclose raising the pH so that it remains constantly above 4.5, and a person of ordinary skill in the art would understand that pH can fluctuate during ethanol production. But the same is true for Veit and Antrim, neither of which teaches lowering the pH after adding phytase.

In fact, the methods disclosed in both pieces of prior art do not appear to require adjusting the pH at all. For example, Veit includes a diagram of the ethanol production process in which beer leaves the fermentation step and proceeds directly to the distillation step, without first going into a beer well or beer mash heat exchanger. Veit, figure 1. U.S. Water contends that this figure inaccurately portrays ethanol plants, and that the missing beer well is a common location for adding sulfuric acid to the beer (to lower pH) before it enters the beer column. Dkt. 226, at 106. But the asserted claims do not require that the beer first travel through the equipment that Veit allegedly omits, and so it is unclear how U.S. Water's disagreement with the figure matters. In fact, Veit's omission of any such machinery merely confirms that the reference does not contemplate adjusting pH between fermentation and distillation. Thus, Veit's disclosure of a pH above 4.5 during fermentation is sufficient to disclose a pH above 4.5 during distillation in the beer column. With Antrim, the disclosure is more specific. Not only is the reference silent on lowering pH after liquefaction, but Antrim purports to eliminate "the need to undesirably adjust the pH level." Antrim, 7:43-45.

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[8] Only paragraph 16 is material to invalidity. The other paragraphs relate to infringement. But they are equally untimely and they, too, will be stricken.

U.S. Water cannot rely on the supplemental validity opinions of Mr. Simms to revive a point that it has already conceded: Veit and Antrim disclose a method wherein the pH of the ethanol processing fluid is 4.5 or higher in the beer column at some point during ethanol production. True, both references disclose pH ranges at earlier points in the ethanol production process. But like the patents-in-suit, neither reference describes or requires adjusting that pH range before beer enters the beer column. Thus, the prior art expressly discloses this element of the asserted claims.

d. Inherent disclosure

The issue on which this case ultimately turns is whether Veit and Antrim inherently disclose using phytase to reduce deposits in ethanol production machinery. Whether a limitation is inherent in the prior art is a question of fact. *SmithKline Beecham Corp. v. Apotex Corp.*, 403 F.3d 1331, 1343 (Fed. Cir. 2005). But as with all questions of fact, if the non-moving party fails to adduce evidence to genuinely dispute that a reference inherently discloses a limitation, then the issue is appropriately decided on summary judgment. *Id.* In the same way that express anticipation requires the prior art to disclose the exact elements of each claim, "inherent anticipation may not be established by probabilities or possibilities . . . the prior art's disclosure [must be] sufficient to show that the natural result flowing from the operation as taught would result in the performance of the questioned function." *King Pharm., Inc.*, 616 F.3d at 1275 (internal citations and quotation marks omitted); *see also Eli Lilly & Co. v. Barr Labs.*, *Inc.*, 251 F.3d 955, 970 (Fed. Cir. 2001) ("A reference includes an inherent characteristic if that characteristic is the 'natural result' flowing from the reference's explicitly explicated limitations."). Here, summary judgment is appropriate because the evidence of record establishes that deposit reduction is a natural result of the methods for adding phytase during ethanol production that Veit and Antrim disclose.

The undisputed evidence establishes that Veit, Antrim, and the patents-in-suit disclose the same chemical process through which phytase breaks down phytic acid. According to one of Novozymes's experts, Dr. Kohl, "[i]n all of its uses, including all of its industrial uses, phytase breaks down phytic acid or salts of phytic acid by the same mechanism of action, namely, cleaving phosphate groups from the inositol core to free the phosphates and produce more soluble products." Dkt. 209, ¶ 23.[9] U.S. Water does not genuinely dispute Dr. Kohl's conclusion.[10] Indeed, the evidence that U.S. Water cites to contradict Dr. Kohl actually *supports* his conclusion. A report authored by one of U.S. Water's own experts, Dr. Reed, explains that "[p]hytase is a generic term used to describe several groups of structurally distinct enzymes that catalyze hydrolysis of phosphomonoester bonds of phytic acid . . . thereby liberating inorganic ortho-phosphate. In order for an enzyme to be a phytase, it must display phosphatase activity." Dkt. 170, ¶ 14. Dr. Reed's report explains that certain environmental variables can affect the amount of phytase activity in ethanol processing. And although those variables can create

[9] A molecule of phytic acid has a core of six carbons, in a ring configuration, and six attached phosphate groups. Dkt. 279, at 11-12. When phytase is present in a solution with phytic acid, the enzyme "cleaves" or "hydrolyzes"—both of which are scientific terms for "separates"—one or more of the phosphate groups attached to the core. *Id.* at 13.

[10] The proposed finding of fact that presented Dr. Kohl's conclusion inadvertently cited to paragraph 24 of Dr. Kohl's affidavit, instead of paragraph 23. Dkt. 210, ¶ 28. Seizing on this oversight, U.S. Water "disputed" the proposed finding of fact on the grounds that Novozymes failed to cite to supporting evidence. Dkt. 235, at 19. The objection is not well-taken, and U.S. Water has not raised a genuine dispute as to this fact.

conditions that limit how much phytic acid the enzyme can break down, Dr. Reed's report does not suggest that *all* phytase activity would stop if the variables are outside optimal ranges.

Rather than disputing that the patents-in-suit teach a chemical reaction involving phytase, U.S. Water contends that the *purpose* of the patents-in-suit is what distinguishes them from the prior art. One of the co-inventors listed in the '244, '137, and '399 patents discussed Veit during his deposition, and he acknowledged that "[t]he basic reaction of phytase with phytic acid is essentially the same in both cases, but we are directing that reaction to a particular goal, which is different from what Veit was using." Dkt. 166 (Young Dep. 306:2-6). According to U.S. Water, this "particular goal" saves the patents-in-suit from being anticipated by the prior art. The court disagrees.

Contrary to U.S. Water's assertion, the differences between the goals of Veit and Antrim and the goals of the patents-in-suit do not preclude the former from anticipating the latter. *See Leggett & Platt, Inc. v. VUTEk, Inc.*, 537 F.3d 1349, 1356 (Fed. Cir. 2008) ("Finally, [the patent holder] repeatedly compares the purpose of the '823 patent to the purpose of the '518 patent, but we fail to see how this comparison proves that the latter's claim is not anticipated by the former's disclosure."). Both sets of patents teach using phytase during ethanol production, in the same dosages and under the same conditions, to perform the same chemical reaction. Even though Veit and Antrim do not expressly identify the benefit that U.S. Water's patents identify, they nevertheless inherently disclose that benefit.

U.S. Water has adduced evidence that one of ordinary skill in the art would not think to practice Veit or Antrim in a way that reduces deposits and, moreover, that practicing Veit and Antrim will not *always* result in deposit reduction. But these considerations (and U.S. Water's supporting evidence) are not relevant because they misunderstand the law of inherent anticipation. "[I]nherency is not necessarily coterminous with knowledge of those of ordinary skill in the art. Artisans of ordinary skill may not recognize the inherent characteristics or functioning of the prior art." *Perricone*, 432 F.3d at 1376 (internal citations and quotation marks omitted); *see also Schering Corp.,* 339 F.3d at 1377 ("[T]his court rejects the contention that inherent anticipation requires recognition in the prior art."). It is therefore immaterial whether one who is skilled in the art would practice Veit or Antrim for the *purpose* of combatting fouling, or even whether the inventors of these patents appreciated every possible result of using phytase to break down phytic acid in ethanol production. Instead, what matters is whether the prior art discloses the conditions that will necessarily result in phytase reducing deposits. And on that issue, there is no genuine dispute. As discussed above, Veit and Antrim expressly disclose each element of U.S. Water's claimed invention.

It is also irrelevant that Veit and Antrim enable benefits other than deposit reduction. The cases that U.S. Water cites to support a contrary conclusion are distinguishable. For example, U.S. Water relies on *MEHL/Biophile Int'l Corp. v. Milgraum*, a case in which the Federal Circuit reviewed a district court's summary judgment that a patent teaching a method for removing hair with a laser was invalid as anticipated. 192 F.3d 1362, 1364 (Fed. Cir. 1999). One of the asserted claims disclosed "aligning a laser light applicator substantially vertically over a hair follicle opening," but the allegedly anticipatory prior reference did not discuss hair follicles or how to align the laser. *Id.* at 1364-65. At issue was whether the reference, a user manual for a tattoo-removing laser, inherently disclosed such a claim. *Id.* at 1365. The Federal Circuit noted that someone who operated a laser as taught by the prior art *could* align it substantially vertically over a hair follicle, but would not necessarily *have* to do so, and on this basis, the court held that the prior art did not inherently anticipate the patent-in-suit. *Id.*

From this holding, U.S. Water quotes that "[o]ccasional results are not inherent." Dkt. 226, at 96 (quoting *MEHL/Biophile Int'l Corp.*, 192 F.3d at 1365). But placed in its appropriate context, the Federal Circuit's statement is not concerned with the "results" of a chemical process or method, like the one at issue in this case. Instead, the court of appeals was referring to whether the allegedly anticipatory prior art adequately described how to perform the *steps* of the claimed invention (*i.e.*, placing the laser substantially vertically over a hair follicle). As this court has already explained, Veit and Antrim expressly disclose the same steps for using phytase that the patents-in-suit claim. Thus, *MEHL/Biophile* does not support the proposition for which U.S. Water cites it.

Indeed, the case does U.S. Water more harm than good. The Federal Circuit went on to hold that a different prior art reference anticipated the patents-in-suit. *MEHL/Biophile*, 192. F.3d at 1366. The reference was a scientific article that described the nature and extent of pigmented cell injury by documenting how laser pulses damaged tissue in guinea pigs. *Id.* at 1364. The court of appeals rejected several arguments that the patent-holders offered against finding that the article anticipated their patent, noting:

> The Polla article concerns itself with guinea pig, rather than human, skin, but that difference is irrelevant to the anticipation analysis. Nothing in the claim limits the method's reach to human skin. Similarly, *the Polla article's failure to mention hair depilation as a goal is similarly irrelevant*. MEHL/Biophile does not dispute on appeal that the laser operating parameters disclosed in the article substantially coincide with those disclosed in the patent. Accordingly, to the extent the embodiment in the patent achieves hair depilation, so does the Polla method. Where, as here, the result is a necessary consequence of what was deliberately intended, it is of no import that the article's authors did not appreciate the results.

Id. at 1366 (emphasis added). *MEHL/Biophile* thus confirms the principle that U.S. Water cannot save the patents-in-suit merely by identifying a new "goal" for the phytase process disclosed by a prior art reference.

Equally distinguishable is the case that U.S. Water cites to support its assertion that "[I]f the teachings of the prior art can be practiced in a way that [is lacking] the allegedly inherent property, the prior art in question does not inherently anticipate." Dkt. 226, at 97 (alterations in original) (quoting *In re Armodafinil Patent Litig. Inc. (%2C722 Patent Litig.)*, 939 F. Supp. 2d 456, 465 (D. Del. 2013), *appeal dismissed* (May 12, 2014), *appeal dismissed* (June 4, 2014), *appeal dismissed* (June 6, 2014)). U.S. Water's quotation includes subtle, but significant wordsmithing. The case actually states that "if the teachings of the prior art can be practiced in a way that *yields a product* lacking the allegedly inherent property, the prior art in question does not inherently anticipate." *In re Armodafinil*, 939 F. Supp. 2d at 465 (emphasis added). Both *In re Armodofinil* and the Federal Circuit precedent that it cited involved patents that claimed chemical compounds, and methods for producing those compounds. *See Glaxo Inc. v. Novopharm Ltd.*, 52 F.3d 1043, 1045 (Fed. Cir. 1995); *In re Armodafinil*, 939 F. Supp. 2d at 460-62.

In re Armodafinil's results-oriented analysis makes sense in its context. If a prior art reference discloses steps that do not always lead to the creation of a chemical compound, then that method might not inherently anticipate a later method that *does* always produce the compound. Indeed, the compound could hardly be a "natural result" of the prior art's disclosure. But that is not the situation presented in this case. Here, U.S. Water's patents do not claim a new product that could only sometimes be produced by practicing Veit or Antrim. The patents-in-suit teach using an already-identified chemical compound, under previously disclosed conditions, to perform a known chemical reaction. Even accepting that U.S. Water has identified a new benefit of that known process, that discovery is not patentable.

Ultimately, the doctrine of inherent anticipation enforces the "basic principle" of patent law that "[t]he public remains free to make, use, or sell prior art compositions or processes, regardless of whether or not they understand their complete makeup or the underlying scientific principles which allow them to operate." *Atlas Powder Co.*, 190 F.3d at 1348. The patents-in-suit, if valid, would prevent the public from practicing Veit and Antrim. U.S. Water emphasizes that the key component of its patents is deposit reduction, and that it is unlikely that anyone practicing Veit or Antrim would "stumble on" this result. Dkt. 226, at 115. But what if an ethanol plant *did* stumble upon a reduction in deposits? U.S. Water's patents, as asserted, would expose the plant to liability for infringement. Indeed, that is the very position that U.S. Water has taken in this case, alleging that several of Novozymes's customers, who practice Veit and Antrim, have infringed on the patents-in-suit because they experienced a reduction in deposits. "[I]t is axiomatic that that which would literally infringe if later anticipates if earlier." *Bristol-Myers Squibb Co.*, 246 F.3d at 1378 (citations omitted). U.S. Water does not persuasively explain how the public could continue to practice Veit and Antrim if the patents-in-suit are valid. Because these references preceded the '137 and '399 patents, they anticipated the invention on which U.S. Water now claims that Novozymes and its customers have infringed.

For all of U.S. Water's allegedly "conflicting" evidence—expert or otherwise—the critical facts that are relevant to the court's anticipation analysis are not in dispute. The patents-in-suit describe a method for using phytase to break down phytic acid; so do Veit and Antrim. The patents-in-suit describe a method for using phytase in terms of ranges for dosage, temperature, and pH; Veit and Antrim disclose the same method, using overlapping and often narrower ranges. According to U.S. Water, the patents-in-suit enable one skilled in the art to practice its claimed invention. Dkt. 204, at 81. Thus, by U.S. Water's own contention, Veit and Antrim are enabling prior art. Because these references expressly or inherently disclose every element of the patents-in-suit, they anticipate the asserted claims in this case.

C. Inequitable conduct

Novozymes accuses the inventors and their attorney of inequitable conduct, based on their failure to disclose information about the *ChemTreat* litigation to the Patent Office during prosecution of the patents-in-suit. U.S. Water has moved for summary judgment that defendants have not adduced sufficient evidence to sustain an inequitable conduct claim. Although the court's decision on validity will end U.S. Water's infringement case, inequitable conduct is still a live issue that could affect Novozymes's entitlement to attorney fees.

To make its inequitable conduct case, Novozymes has the burden to show, by clear and convincing evidence, that the patentees failed to disclose material information during prosecution with the specific intent of deceiving the Patent Office into issuing the patent. *Therasense, Inc. v. Becton, Dickinson & Co.*, 649 F.3d 1276, 1290 (Fed. Cir. 2011). It is a high standard on both elements. The information must be *but-for* material, which means that the patent would not have issued if the information had been disclosed to the examiner. *Id.* at 1291. And although intent can be, and usually is, shown by circumstantial evidence, the evidence must make the intent to deceive the single most reasonable inference. *Id.* at 1290.

Novozymes's basic theory is that in the course of the *ChemTreat* litigation, U.S. Water pressed for a broad interpretation of the '244 patent, the parent to the patents-in-suit. But the broad interpretation did not fly with the *ChemTreat* court, which limited the '244 patent to processes that added phytase after fermentation, based on a prosecution history estoppel arising from the patentees' characterization of the scope of the '244 patent and the prior art. The narrow construction doomed U.S. Water's infringement case. The applications for the patents-in-suit were then pending as continuations of the '244 patent's application. U.S. Water, learning from its failure in the *ChemTreat* case, realized that it would need broader claims if it wanted to succeed in future infringement cases. So the patentees amended the claims in the pending applications to remove the limitation that phytase be added after fermentation. But they did not say a word about the *ChemTreat* case to the Patent Office, even though the litigation was fresh in their minds and they had the opportunity to do so. As Novozymes rightly points out, the patentee has the duty to disclose litigation involving the subject matter of a pending application. M.P.E.P. § 2001.06.

The cornerstone of Novozymes's inequitable conduct case is a July 23, 2012, amendment, in which the patentees removed the limitation that phytase be added after fermentation. In presenting the amended claim, so Novozymes's argument goes, the patentees mischaracterized the amended claim as being "generally patterned on issued claim 1" of the '244 patent. According to Novozymes, the patentees knew well that the amended claim was not patterned on claim 1 of the '244 patent, because the *ChemTreat* judge had just made it clear that the '244 patent was limited to processes in which phytase was added after fermentation. If the patentees had set all this out for the examiner, the examiner would have forced patentees to confront all of their previous statements, including their arguments about the prior art, and the patents-in-suit would not have issued because the *ChemTreat* information would have made clear to the examiner that the broader claims could not issue over Veit and Caransa, another reference at issue during prosecution.

Novozymes has shown that U.S. Water was willing to advance some questionable claim construction positions in the *ChemTreat* litigation concerning the scope of the '244 patent. But Novozymes falls short of establishing the but-for materiality of the *ChemTreat* information in the prosecution of the patents-in-suit. The July 23, 2012, amendment set out the amended claim with differences with claim 1 of the '244 patent highlighted. It would have been amply clear to the examiner that the patentees were seeking a claim that was broader in the sense that the after-fermentation limitation was removed. Novozymes is correct that the third-party submission concerning the amended claims does not relieve the patentees of their own duties of candor. But that third-party submission provides one more reason that the examiner knew she had to evaluate the allowability of the broader claims over the prior art, particularly Veit and Caransa. On this record, there is simply no evidence that information about the arguments made during the *ChemTreat* litigation, or any decision from the *ChemTreat* court, would have had any impact on the claims of the patents-in-suit. Novozymes's argument is, essentially, that without the *ChemTreat* information, the examiner just could not understand the amended claims well enough to do her job. But the examiner was fully aware that the broader claims had to be examined over Veit and Caransa, and a general allegation that the examiner did not pay attention to what was put before her is not enough to sustain an allegation of inequitable conduct. *Fiskars, Inc. v. Hunt Mfg. Co.*, 221 F.3d 1318, 1327 (Fed. Cir. 2000) ("An applicant is not required to tell the PTO twice about the same prior art, on pain of loss of the patent for inequitable conduct.").

Novozymes also falls short of showing deceptive intent. Novozymes shows that the patentees recognized the benefit of securing broader claims in the continuation applications, and thus the patentees had a motive to lie to the Patent Office. But the desire to secure broader claims in a continuation is an utterly routine situation in patent prosecution. Novozymes has no evidence that would make the inference of deceptive intent the single most reasonable inference to be drawn from the patentees' failure to disclose the *ChemTreat* litigation. The inventors and their attorney could have made the reasonable determination that their unsuccessful attempts to broaden the scope of the '244 patent in litigation were not material to their overt attempts to secure broader claims in the continuations. Nor can Novozymes make out a case of inequitable conduct on the basis of U.S. Water's arguments to the Patent Office about the content of the prior art, when that prior art was before the examiner. *Innogenetics, N.V. v. Abbott Labs.*, 512 F.3d 1363, 1379 (Fed. Cir. 2008).

In sum, defendants have failed to adduce evidence sufficient to establish either materiality or deceptive intent. Plaintiff's motion for summary judgment will, in this respect only, be granted.

CONCLUSION

The court's decision that the asserted claims of the patents-in-suit are anticipated and that the defendants have not adduced evidence sufficient to sustain their burden to show inequitable conduct disposes of this case. The court will not reach the issues of whether the patents-in-suit are infringed, or whether those patents are invalid on any other grounds.

All other pending motions will be denied as moot, and the clerk of court will close this case.

ORDER

IT IS ORDERED that:

1. Novozymes's motion to strike certain paragraphs of the Simms declaration, Dkt. 273, is GRANTED.

2. Defendants' motion for summary judgment that claims 1, 6, 12, and 13 of the '137 patent, and claims 1, 2, 5-12, 16-22, 25, 28-32, 34, and 35 of the '399 patent are invalid, Dkt. 199, is GRANTED.

3. Defendants' motion is in all other respects DENIED as moot.

4. Plaintiffs' motion for summary judgment on the issue of inequitable conduct, Dkt. 182, is GRANTED.

5. Plaintiffs' motion is in all other respects DENIED as moot.

6. All other pending motions are DENIED as moot.

Entered July 29, 2015.

BY THE COURT:

/s/

JAMES D. PETERSON
District Judge

Addendum 3: U.S. Patent No. 8,415,137

U 7499057

THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

October 07, 2014

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THIS OFFICE OF:

U.S. PATENT: *8,415,137*

ISSUE DATE: *April 09, 2013*

By Authority of the

Under Secretary of Commerce for Intellectual Property and Director of the United States Patent and Trademark Office

P. SWAIN

Certifying Officer

US008415137B2

(12) **United States Patent**
(10) **Patent No.: US 8,415,137 B2**
(45) **Date of Patent: *Apr. 9, 2013**

Johnson et al.

(54) **PREVENTING PHYTATE SALT DEPOSITION IN POLAR SOLVENT SYSTEMS**

(75) Inventors: **Roy Johnson**, Oconomowoc, WI (US); **Paul R. Young**, Wheaton, IL (US)

(73) Assignee: **U.S. Water Services, Inc.**, St. Michael, MN (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **13/274,075**

(22) Filed: **Oct. 14, 2011**

(65) **Prior Publication Data**

US 2012/0093979 A1 Apr. 19, 2012

Related U.S. Application Data

(63) Continuation of application No. 11/873,630, filed on Oct. 17, 2007, now Pat. No. 8,039,244.

(51) **Int. Cl.**
D06M 16/00 (2006.01)

(52) **U.S. Cl.** **435/264**; 134/18; 134/22.19

(58) **Field of Classification Search** .... None
See application file for complete search history.

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(57) **ABSTRACT**

The present inventors have surprisingly discovered that phytic acid tenaciously precipitates with soluble metals in food or fuel ethanol-processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting phytic acid salts or phytates to inorganic phosphates to improve metal solubility and reduce deposition within processing equipment.

13 Claims, No Drawings

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PREVENTING PHYTATE SALT DEPOSITION IN POLAR SOLVENT SYSTEMS

This application is a Continuation application of U.S. Ser. No. 11/873,630, filed Oct. 17, 2007, and which application is incorporated herein by reference. A claim of priority, to the extent appropriate, is made.

FIELD OF THE INVENTION

The present inventors have surprisingly discovered that the phytic acid tenaciously precipitates with soluble metals in food or fuel ethanol-processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting phytic acid salts or phytates to inorganic phosphates to improve metal solubility and reduce deposition within processing equipment.

BACKGROUND

Fermentation of sugars and polysaccharides into alcohol is a rapidly developing technology for producing liquid fuel, such as gasohol or E85, which are the most common examples in the United States and contain varying amounts of ethanol and gasoline. Billions of gallons of fuel ethanol are produced every year through the fermentation of grains, plants and feedstock, primarily corn. Other types of feedstock such as sugar care and cellulose are also increasing in importance.

Ethanol producers have found scale deposits on processing equipment at several stages of ethanol processing. These scale deposits are known to impede heat transfer and flow, and interfere with the proper operation of mechanical devices used in ethanol processing. The deposits tend to be most severe or tenacious on hot surfaces, and where the pH of the processing liquid is highest (about 4.5), but deposits may also form at lower pH values and on cooler surfaces. There remains a need for methods and compositions for reducing this scale formation.

SUMMARY

The present inventors have unexpectedly discovered that phytic acid tenaciously precipitates with soluble metals in food or fuel ethanol-processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting phytic acid salts or phytates to inorganic phosphates to improve metal solubility and reduce deposition within processing equipment.

In an embodiment, the present method can reduce insoluble deposit formation in equipment that contacts food or fuel ethanol-processing fluids. The method can include: adding an agent to the ethanol-processing fluids after fermentation; converting the insoluble material to a soluble residue by action of the agent; and removing the soluble residue from the equipment that contacts the ethanol-processing fluids. The method can also include identifying the insoluble deposit from the ethanol-processing fluids.

In an embodiment, the present method includes adding an enzyme with phytase activity to the ethanol-processing fluids after fermentation; converting the phytate to orthophosphate by action of the enzyme; and removing the soluble orthophosphate from the equipment that contacts the ethanol-processing fluids. This embodiment can also include identifying the insoluble deposit from the ethanol-processing fluids as phytic acid or a salt of phytic acid.

DETAILED DESCRIPTION

Definitions

As used herein, the term "mash" refers to a mixture or slurry of milled grain, process water and an enzyme such as alpha amylase, after the mixture has been subjected to a high temperature pressure "cook" and introduced into fermentation tank during ethanol processing.

"Cook water" refers to process water generated and/or used during cook process, where the starch content of milled grain is physically and chemically prepared for fermentation, typically by application of heat and by the action of enzymes such as amylase.

"Liquefaction" or "liquefy" means a process by which starch is converted to shorter chain and less viscous dextrins. Generally, this process involves gelatinization of starch simultaneously with or followed by the addition of enzymes such as amylase.

The term "liquefaction slurry" refers to combination of hot slurry, and the slurries from primary and secondary liquefaction produced during ethanol processing. Hot slurry is formed when milled grain is first mixed with process water and the formed slurry is treated with an enzyme such as α-amylase and then heated to temperatures of up to 190° F. to reduce the viscosity of the slurry. The slurry is then pumped through a pressurized jet cooker for flash condensation during primary liquefaction. After flash condensation and cooling, the primary liquefaction slurry is held at high temperature for one to two hours to provide enough time for the amylase to fully break down the starch into short chain dextrins. During secondary liquefaction or "saccharification", a second enzyme (such as glucoamylase) is added, and the formed slurry is moved into fermentation tanks Liquefaction and saccharification may take place successively or simultaneously.

"Fermentation" refers to a process by which the sugars in the slurry or mash from liquefaction/saccharification are converted into alcohol by the action of yeast in the fermentation tanks or fermentors. The mash is allowed to ferment for 50-60 hours, resulting in a mixture that contains about 15% ethanol as well as the solids from the grain and added yeast, i.e. the "fermentation slurry." Once fermentation is complete, the mash or slurry is called "beer" and is moved into beer wells to be used for ethanol distillation and recovery.

The term "whole stillage" refers to the mash or solids remaining after ethanol is removed from beer or beer mash using a stripper column. The term is used interchangeably with the term "thick stillage." Whole stillage is typically 11% to 14% solids and contains all of the other non-starch components of the grains that pass through the process (germ, protein, gluten, hull & fiber etc.).

"Thin stillage" refers to the liquid removed from the mash in ethanol production. Thin stillage is about 5% dry matter and about 95% water. Thin stillage can be reintroduced into the cooking and distillation processes to extract additional ethanol. Thin stillage that is recycled to the beginning of the dry-grind process is known as "backset" and is used to conserve water used in processing.

The term "beerstone" refers to a hard organometallic scale deposited on fermentation equipment and that is primarily calcium oxalate. Although beerstone is a commonly formed deposit during ethanol processing, not all solid deposits formed are beerstone.

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The term "phytase unit" refers to the amount of phytase enzyme that can liberate one micromole of ortho-phosphate from insoluble phytate in one minute, assuming optimal conditions of temperature and pH.

Methods of the Invention

The commercial processing of ethanol produces aqueous slurries of plant grains and fibers that release phytic acid. The present inventors have unexpectedly discovered that the phytic acid tenaciously precipitates with soluble metals in the processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting insoluble phytic acid salts (i.e., phytate) to soluble inorganic phosphates and an organic compound (i.e., inositol), which can improve metal solubility and reduce deposition within processing equipment.

The present invention provides a method for reducing or even preventing the formation of insoluble material, deposits, or scale on equipment used in processing of food or fuel ethanol. According to the method, the deposit or the material forming the deposit can be converted into a soluble material by the action of an agent capable of degrading or breaking down the insoluble deposit or material that forms the deposit. The soluble material can then be easily removed from the equipment or processing system, by standard methods or conventional means known to those of skill in the art. The method can include identifying content of the deposit.

In an embodiment, the method of the present invention includes reducing the formation of insoluble deposits during ethanol processing. During commercial food and fuel ethanol processing, the aqueous slurries of plant grains and fibers produce acidic residues that interact with soluble metals in ethanol-processing fluids to produce organometallic precipitates. Many of these precipitates are insoluble solids that deposit as scale on processing equipment and interfere with downstream processing of ethanol, by impairing heat transfer and causing production interruptions. In an aspect, the solid deposit formed in this manner is beerstone, composed primarily of calcium oxalate. In another aspect, the aqueous slurries of plant grains and fibers form phosphate salts with dissolved metals present in ethanol processing fluid, such as salts of magnesium or calcium phosphate, for example. Various types of phosphate salts can be formed during ethanol processing including, without limitation, newberyite, bobierite, struvite (Mg salts), brushite, fluorapatite, hydroxyapatite (Ca salts), etc. In an aspect, the phosphate salt is a salt of a dissolved metal and phytic acid ($C_6H_{18}O_{24}P_6$; myoinositol hexakisphosphate, a phosphate ester of inositol) that is released by the plant grains and fibers present in the aqueous slurries. In another aspect, the phosphate salt is magnesium phytate. Phytate salts have been shown to form tenacious or insoluble precipitates in the presence of polar protic solvents such as water and ethanol, both of which are present in various concentrations during ethanol processing. Therefore, in an aspect, the method of the present invention provides for reducing or removing phytate that can deposit on ethanol processing equipment.

In an embodiment, the method of the present invention includes identifying the insoluble material formed during ethanol processing. Many different organometallic salts may be formed by the plant grains and fibers present in ethanol processing fluids. The solubility products of each salt may vary with processing conditions such as temperature and pH. Accordingly, the method can include chemically and/or geologically identifying the deposit or materials susceptible to deposit, which can aid in reducing or removing the insoluble deposit from the processing equipment and processing fluids. The insoluble material may be identified by standard methods known to those of skill in the art, including dry analysis methods such as x-ray fluorescence (XRF) or oxidation, followed by elemental analysis, for example, or wet analysis methods such as acid-base neutralization reactions, for example. In an aspect, the insoluble material is identified as a phosphate salt. In another aspect, the insoluble material is identified as a phytate salt, and in yet another aspect, the insoluble deposit is identified as magnesium phytate.

A considerable amount of the phosphorus or phosphate content in plant grains and fibers is in the form of phytic acid (as identified by standard analysis methods such as high temperature sample oxidation to ash), and commercial processing of these plant grains and fibers leads to release of phytic acid. These phytic acid concentrations in liquids, such as ethanol-processing fluids, can be high enough to cause precipitation of metal phytate salts, such as magnesium phytate, and subsequent deposit formation, in ethanol processing equipment. The formed phytates can impair heat transfer and cause production interruptions. A small amount of phytic acid is naturally broken down into soluble byproducts (i.e., soluble phosphates) during fermentation, but a large quantity (i.e., approximately 30-35%) of the phosphorus or phosphate in the stillage and syrup remains as phytic acid or phytate.

Metal phytate salts are generally much less soluble than the corresponding metal phosphates. For example, magnesium phytate is more than an order of magnitude less soluble than magnesium phosphate, and therefore, tends to precipitate out more readily than the more soluble magnesium phosphate. One way of causing phytate to precipitate is to heat a stable solution of magnesium phytate. Because phytate is less soluble at higher temperatures, a temperature is eventually reached where precipitation occurs, and this temperature is lower than the temperature at which magnesium phosphate would precipitate out. The temperature at which precipitation occurs is a function of pH and concentration of magnesium and phosphate or phytate ions. Assuming similar pH conditions, a solution of magnesium phosphate must be heated to about 40° C. more than a magnesium phytate solution, in order for the phosphate salt to precipitate out, even where the concentration of magnesium and phosphate ions were far greater than the concentration of magnesium and phytate ions, as shown in Table 1:

TABLE 1

Solubility of Magnesium with Phosphate and Phytate

Solution	pH	Temp (° C.)	Precipitate (+/−)
2100 ppm Mg^{2+} 8400 ppm PO_4^{3-} (as phosphate)	5.2	40	−
	5.2	60	−
	5.2	90	+
	5.76	60	−
	5.76	80	++
800 ppm Mg^{2+} 3600 ppm PO_4^{3-} (as phytate)	4.23	80	−
	4.52	60	−
	4.52	80	+
	4.97	40	−
	4.97	60	++
	5.36	21	−
	5.36	40	++
	5.6	21	+

Furthermore, the presence of ethanol also reduces the solubility of magnesium phytate. Table 2 indicates that precipita-

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tion of the phytate salt occurred at much lower temperature in the presence of ethanol, even at very similar pH. In the table, the water and ethanol columns indicate the temperature at which a precipitate became visible in a solution containing 800 ppm Mg^{2+} and 3600 ppm phytic acid.

TABLE 2

Solubility in Presence of Ethanol

pH	Temperature (° C.; in 100% water solution)	Temperature (° C.; in 13% ethanol/87% water solution)
4.35		40
4.52	80	
4.53		30
4.6		21
4.97	60	
5.36	40	
5.6	21	

In an embodiment, the present invention provides a method for reducing or removing insoluble material, such as phytate precipitates, by the action of an agent added to ethanol processing fluids. In an aspect, the agent is an acidic compound that can break down organic phosphates and phosphonates into soluble inorganic phosphates in the presence of a strong oxidizer or oxidizing agent. For example, a persulfate can degrade the insoluble phytate through acid digestion. In another aspect, the agent is an acidic compound that, in combination with ultraviolet light, can break down organic phosphates and phosphonates into soluble inorganic phosphates. In an embodiment, the agent is an enzyme capable of digesting or degrading (e.g., hydrolyzing) organic phosphates or phosphonates into soluble inorganic phosphates and an organic compound. For example, the agent can be a phytase, which can hydrolyze phytate to inorganic phosphate and inositol.

Phytase is an enzyme known to be capable of breaking down the phytic acid found in plant material. It is currently used primarily in animal feed applications, where it helps convert insoluble organic phosphates into soluble phosphorus that is more readily available to the animal's digestive system, and thereby also reduces environmental contamination by insoluble phosphate salts such as phytates. In ethanol processing, the phytase has been used to increase the bioavailability of phosphorus for the action of yeast in pre-saccharification and fermentation. Similarly, phytase has also been used in the liquefaction stage, or prior to fermentation to improve the activity of α-amylase.

Phytase is commercially available and can be derived from a variety of sources. In an aspect, the phytase is obtained from plants or microorganisms, such as bacteria, or from fungi, such as yeast or filamentous fungi, as disclosed in U.S. Patent Pub. No. 20050272137, for example, and incorporated herein by reference. Plant phytases may be derived from wheat-bran, maize, soybean, or lily pollen. Bacterial phytases may be derived from various bacterial sources including, without limitation, *Bacillus*, *Pseudomonas*, or *Escherichia*, preferably *B. subtilis* or *E. coli*. In another aspect, the phytase is a yeast phytase derived from *Saccharomyces* or *Schwanniomyces*, preferably *Saccharomyces cerevisiae* or *Schwanniomyces occidentalis*. In yet another aspect, phytases may be derived from filamentous fungi, including, but not limited to, species from the genus *Aspergillus, Thermomyces, Myceliophthora, Manascus, Penicillium, Peniophora, Agrocybe*, and the like.

Suitable commercially available phytases include, without limitation, those sold under the tradenames MAXALIQ™ ONE, by Genencor (Beloit, Wis.), RONOZYME® P5000 by Novozymes (Denmark), PHYTASE 5000L by DSM Food Specialties (France), NATUPHOS® 5000 by BASF (Germany), and PHYZYME™ XP 10000 by Danisco Animal Nutrition (St. Louis, Mo.). Suitable commercially available phytase enzyme may also be obtained from suppliers including, without limitation, Deerland Enzymes (Kennesaw, Ga.).

In an embodiment, phytase is added to ethanol processing equipment and/or processing fluid at a time point and under conditions required for the particular type of equipment or stage of ethanol processing. In an aspect, phytase is added to fermentation fluid to reduce formation of insoluble deposits in downstream ethanol processing equipment. In another aspect, phytase is added to ethanol processing fluids downstream of the fermentation process, such as, for example, beer, whole stillage, thin stillage, backset, centrate, or a mixture of these fluids. In an embodiment, phytase is added to thin stillage or backset. In an aspect, phytase is added to thin stillage or backset in line, i.e. the enzyme is introduced directly into thin stillage or backset-containing equipment during ethanol processing. In another aspect, phytase is added to thin stillage or backset offline, i.e. the enzyme is added to thin stillage or backset contained in a separate vessel or tank. Phytase-treated thin stillage or backset can then be cycled back into processing lines from the vessel or tank.

In an embodiment, the present invention provides a method in which the agent or enzyme is introduced into the ethanol-processing fluid under optimal conditions of temperature and pressure. Where the agent is phytase, the term "optimal conditions" refers to those conditions of concentration, temperature, residence time or reaction time, and pH that allow sufficient reaction with soluble phytate, phytate suspension, phytate precipitate, or insoluble phytate scale that reduces the level of the phytase deposit to an amount acceptable for operation of the ethanol plant or process. In an embodiment, the conditions provide for complete hydrolysis of soluble phytate and phytate suspension.

In an aspect, the phytase is added to the ethanol processing fluid at temperatures of about 20° C. to about 80° C., for example, about 20° C. to about 77° C., about 40° C. to about 65° C., or about 30° C. to about 55° C. (e.g., 52° C.). In an aspect, the phytase is added to the ethanol processing fluid at temperatures sufficient to allow the reaction between phytate and phytase to proceed to completion without degrading the enzyme. In another aspect, the phytase is added to the ethanol processing fluid at pH of about 3 to about 9, for example, about 4.0 to about 5.0, about 4.0 to about 5.5, or about 4.0 to about 5.3. In yet another aspect, the phytase is added at a pH of 4.0, and the reaction is conducted at temperatures of about 40° C. to about 65° C., about 20° C. to about 77° C., or about 30° C. to about 55° C. (e.g., 52° C.).

In an aspect, the phytase is added to the ethanol processing fluids at a concentration of about 100 ppm to about 500 ppm. In another aspect, the phytase is added at a concentration of 100 ppm, and in yet another aspect, the phytase is added at a concentration of 500 ppm. The phytase can be added at concentrations expressed in phytase units. A unit of activity (U) is the amount of phytase that can release 1 μmol of orthophosphate per minute from excess phytic acid/phytate, at a temperature of 37° C. and a pH of about 5.5. Therefore, in an aspect, the phytase is added at a concentration of about 500 U/L to about 2500 U/L. In another aspect, the phytase is added at a concentration of 100 U/L, and in yet another aspect, the phytase is added at a concentration of 2500 U/L.

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In an embodiment, the phytase is added to ethanol processing fluids at lower concentrations, and the reaction is allowed to proceed over longer periods of time. Extending the reaction time or residence time allows smaller amounts of enzymes to be used, making ethanol processing more economical. In an aspect, the phytase is added to the ethanol processing fluids for a residence time sufficient for complete reaction of the phytase with the insoluble phytate. In another aspect, the phytase is added for a residence time of about 2 minutes to about 1200 minutes, for example, about 3 minutes to about 200 minutes, or about 3 minutes to about 40 minutes.

As shown in Table 3, low concentrations of phytase can release significant amounts of ortho-phosphate, if reacted over longer periods of time. For example, 5 ppm of phytase will release approximately 900 ppm of ortho-phosphate when reacted over a 20 hour time period. In contrast, 100 ppm of phytase releases 400-500 ppm of ortho-phosphate in just 10 minutes.

TABLE 3

Orthophosphate Formation at Low Doses of Phytase

	Amount of Released Ortho-Phosphate (ppm)		
Time (min)	5 ppm phytase	10 ppm phytase	20 ppm phytase
20	73	92	109
60	143	232	387
140	260	436	732
260	377	636	933
1200	894	1460	1713

Therefore, in an aspect, the phytase is added at a concentration of about 5 ppm to about 20 ppm. In another aspect, the phytase is added at a concentration of 5 ppm. In a further aspect, the phytase is added at a concentration of 10 ppm, and in a yet further aspect, the phytase is added at a concentration of 20 ppm. These concentrations may also be expressed in phytase units such that, in an aspect, the phytase is added at a concentration of about 25 U/L to about 100 U/L. In another aspect, the phytase is added at a concentration of 25 U/L. In a further aspect, the phytase is added at a concentration of 50 U/L and in a yet further aspect, the phytase is added at a concentration of 100 U/L.

In an embodiment, the present invention provides a method in which the agent added to the ethanol processing fluid converts soluble phytate, phytate suspension, phytate precipitate, or phytate scale into soluble orthophosphate. In an aspect, the agent is an enzyme such as phytase, which specifically hydrolyzes phytate. The enzyme can be used to convert phytate materials into soluble phosphates that are easily removed from the processing fluid, if necessary. Assuming identical conditions of pH (5.3) and identical concentrations of Mg^{2+} ions (1048 ppm) and phytic acid (4702 ppm), samples of processing fluid treated with phytase remained clear (i.e., phosphates remain in solution without precipitating out). However, samples of processing fluid not exposed to phytase showed progressively more precipitation of magnesium phytate as the temperature is increased, as shown in Table 3 below.

TABLE 4

Enzyme Treatment Prevents Precipitation

Enzyme Treated? (Y/N)	Temp (° C.)	Precipitate? (+/-)
Y	20	-
Y	40	-
Y	80	-
Y	100	-
N	20	+
N	40	++
N	60	+
N	80	+++

The following examples are provided to illustrate various aspects of the invention, and should not be construed to limit the invention. A person of skill in the art will recognize that various modifications may be made to the examples without departing from the scope of the present invention.

EXAMPLES

Example 1

Conventional Analyses Indicate that Deposits in the Beer Column Contain Phosphorus, But Do Not Reveal That the Phosphorus is in Phytate

The content of various solid deposits formed during ethanol fermentation can be determined using standard methods. It was previously thought that the solid deposits in the beer column were primarily beerstone (i.e. calcium oxalate). The unexpected results shown in the following examples demonstrate, however, that a large percentage of the solid deposits found in the beer column are in the form of phosphates, i.e. P_2O_5.

Materials and Methods

To determine the content of the solid deposit in the beer column, X-ray fluorescence (XRF) analysis was used. A sample of solid deposit from the beer column was collected and air-dried. A portion of the sample was ground to approximately 400 mesh using a steel swing mill, and the ground sample was analyzed by XRF. Using standard XRF procedures, it was possible to determine the presence of 31 major, minor and trace elements to a relative precision/accuracy of approximately 5-10% for major and minor elements and approximately 10-15% for trace elements. A replicate sample was analyzed, along with a standard reference material ("SY3", a CANMET standard rock or geological sample) to demonstrate analytical reproducibility as well as analytical accuracy for a geological standard.

Results

The content of the solid deposit in the beer column was as shown in Table 5. Major elements in the solid deposit include magnesium (as MgO) and phosphorus (as P_2O_5), while potassium and calcium (as their respective oxides K_2O and CaO) occur as minor elements. Trace amounts of zinc were also detected. Major and minor elements were represented as weight percentages of the deposit, while trace elements were represented in ppm units.

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TABLE 5

Deposit Analysis	
Element	Content
Magnesium (as MgO)	12.5 wt %
Phosphorus (as P_2O_5)	35.7 wt %
Potassium (as K_2O)	2.54 wt %
Calcium (as CaO)	2.76
Zinc	18447 ppm

Conclusions

The results in Table 5 demonstrate that the majority of the solid deposits are present as phosphate (rather than as the expected beerstone or calcium oxalate). XRF analysis of solid deposits shows a large concentration of magnesium and phosphorus present in the oxide form, but does not distinguish between different chemical forms of phosphorus or phosphate salts, and does not specify if some of the magnesium was actually present as a phosphate salt (i.e. magnesium phytate). Because different forms of phosphorus and phosphate salts have different solubilities and because the phytase has different activities on different forms of phosphate, it is useful to distinguish between the various forms.

Example 2

The Deposits Include Substantial Amounts Phosphorus in Phytate

Because the activity of the phytase on the solid deposits is dependent on the type of phosphorus or phosphate salts present, wet analysis of various deposit samples was used to determine the different forms of phosphorus and phosphate salts present in the solid deposits of the beer column.

Materials and Methods

Deposit samples from ethanol plants were dissolved in a weighed amount of acid, the acid was neutralized and the solution was diluted to a known volume. A portion of the neutralized sample was then subjected to a standard test for ortho-phosphate (PO_4^{3-}), i.e. the Hach test. Total phosphorus was determined by acid oxidation with persulfate, followed by the reactive phosphorus test. Organically bound ortho phosphate was then determined by subtracting the acid-hydrolysable phosphorus content, and was reported as PO_4^{3-} or ortho-phosphate.

A separate portion of the neutralized solution was treated with phytase (at a concentration of 500 ppm and 41° C. for 20-30 minutes). Under these conditions, phytate present in the solution was converted to ortho-phosphate. The amount of ortho-phosphate (PO_4^{3-}) present after phytase treatment represents the total phosphorus content of the deposit (total P as PO_4^{3-}), i.e. original ortho phosphate and phytate phosphorus.

Results

As indicated in Table 6, deposit samples from an evaporator at one plant, and samples from cook water lines and liquefaction pumps at a second plant had between 60% and 75% of their phosphorus content as phytate, confirming that the deposit in the beer column included primarily magnesium phytate.

TABLE 6

Comparison of Deposit Analysis by XRF and Wet Methods

	Content by XRF		Content by Wet Analysis	
Sample	% MgO	% P_2O_5	% PO_4^{3-}	Total % P as PO_4^{3-}
Plant 1 Evaporator (1)	14.6	36.8	7.4	37.4
Plant 1 Evaporator (2)	11.3	23.0	1.9	27.7
Plant 1 Evaporator (3)	10.5	25.5	5.9	30.9
Plant 2 Cook Water	14.0	35.5	2.4	36.0
Plant 2 Pump	16.7	35.2	9.1	38.0

Conclusions

The results in Table 6 demonstrate that the solid deposits were present as phytate salt, i.e. magnesium phytate. This is an important discovery because the activity of the phytase added to various structures in an ethanol plant depends on the presence of phosphate primarily in the phytate form. Solid deposits that exist primarily as phytate can be dissolved by the action of the phytase.

Example 3

Phytase Reduces the Phytate Concentration in Thin Stillage, Which is the Source of the Deposits

Materials and Methods

The amount of phosphorus (present as ortho phosphate) in the thin stillage fraction collected from an ethanol plant will decrease once phytase has been added. To determine the decrease in ortho-phosphate concentration over time as a result of enzyme activity, thin stillage fractions were collected and treated with phytase at temperatures from 43° C. to 62° C., at a pH of 4.0. The phytase concentration was either 100 ppm or 500 ppm. After adding the enzyme, ortho-phosphate concentration was measured at various time points (e.g., from 3 minutes to 20 minutes). Any phosphorus present as phytate (about 40-50% of the total phosphate) was converted to ortho phosphate (PO_4^{3-}) by the action of the phytase, with more phytate converted over increasing periods of time. The concentration of ortho-phosphate in the thin stillage was therefore a measure of enzyme activity.

Results

As demonstrated in Table 7, at an enzyme concentration of 500 ppm and a temperature of 43° C., the reaction was essentially complete in 5 minutes, indicating that phytate was completely converted to ortho phosphate. At an enzyme concentration of 100 ppm, and a temperature of 62° C., the reaction continued for 20 minutes.

TABLE 7

Conversion of Phytate in Thin Stillage to Ortho Phosphate

Sample/Run (min)	Enzyme Conc. (ppm)	Temp (° C.)	Phosphate Conc. (ppm)	Released PO_4^{3-} Conc. (ppm)
Distilled Water (zero)	—	—	0	—
Std. 1 - 640 ppm PO_4^{3-}	—	—	732	—
Std. 2 - 1280 ppm PO_4^{3-}	—	—	1234	—

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TABLE 7-continued

Conversion of Phytate in Thin Stillage to Ortho Phosphate

Sample/Run (min)	Enzyme Conc. (ppm)	Temp (° C.)	Phosphate Conc. (ppm)	Released PO_4^{3-} Conc. (ppm)
Run 1				
5 min	500	45.5	3313	1463
10 min	500	43.3	3385	1536
Std 2			1242	—
Run 2				
3 min	100	63.3	1688	137
5 min	100	62.8	2146	595
10 min	100	62.2	2214	663
20 min	100	61.0	2859	1309

Conclusions

The results shown in Table 7 indicate that substantially all of the phosphorus present in the phytate form was converted and released as ortho-phosphate by the action of the phytase. About half of the total phosphorus content of the thin stillage was in the form of phytate, and complete reaction of the enzyme with the phosphorus resulted in the conversion of phytate and release of ortho-phosphate. The amount or concentration of released ortho-phosphate therefore provides a measure of the activity of the phytase.

Example 4

Increased Solubility on Conversion of Phytate to Ortho-phosphate

The solid deposits formed in various structures in an ethanol plant include primarily insoluble magnesium phosphate salts, including magnesium phytate. The following example illustrates that the solubility of these salts is unexpectedly increased in the presence of phytase.

Materials and Methods

To determine the effect of phytase on increasing the solubility of magnesium phytate, two solutions of magnesium phytate were prepared by mixing magnesium salt with phytic acid in two separate test tubes. The solution in one test tube was treated with phytase, while the other was left untreated. The two solutions were maintained at the same temperature and pH and had identical magnesium and total phosphorus content.

Results

The action of the phytase on magnesium phytate converted the insoluble phytate salt into soluble ortho phosphate, which can then be readily removed from pumps, lines and evaporators in an ethanol plant. FIG. 1 illustrates the differences in solubility of phytic acid solutions with and without the action of phytase.

The enzyme-treated solution in one test tube remained clear, indicating that all the phytate was converted to soluble ortho phosphate. The untreated solution showed precipitate formation, indicating the presence of insoluble magnesium phytate. This demonstrated that the enzyme completely converted the phytate to ortho-phosphate and greatly increased the solubility of the magnesium salt.

Conclusion

Phytase can prevent solid deposits of magnesium phosphate salts from forming in various structures in an ethanol plant.

Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art. Although any methods, devices and material similar or equivalent to those described herein can be used in practice or testing, the methods, devices and materials are now described.

All publications and patent applications in this specification are indicative of the level of ordinary skill in the art to which this invention pertains and are incorporated herein by reference in their entireties.

In this specification and the appended claims, the singular forms "a," "an," and "the" include plural reference, unless the context clearly dictates otherwise. Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood to one of ordinary skill in the art.

The various embodiments described above are provided by way of illustration only and should not be construed to limit the invention. Those skilled in the art will readily recognize various modifications and changes that may be made to the present invention without following the example embodiments and applications illustrated and described herein, and without departing from the true spirit and scope of the present invention without following the example embodiments and applications illustrated and described herein, and without departing from the true spirit and scope of the present invention, which is set forth in the following claims.

The claimed invention is:

1. A method of reducing formation of insoluble deposits of phytic acid or salts of phytic acid on surfaces in a fuel ethanol-processing equipment, the method comprising:

adding phytase to an ethanol processing fluid in the equipment containing phytic acid or salts of phytic acid under conditions suitable for converting the insoluble phytic acid or phytic acid salts to soluble products; thereby reducing the formation of deposits of insoluble phytic acid or phytic acid salts on surfaces in the equipment; wherein the equipment in which deposit formation is reduced comprises a beer column, and

wherein the pH of the ethanol processing fluid in the beer column is 4.5 or higher during production of ethanol.

2. The method of claim **1**, wherein converting comprises partial conversion of phytate to phosphate and inositol.

3. The method of claim **2**, wherein converting further comprises reacting the insoluble phytic acid salts in the presence of polar protic solvents.

4. The method of claim **3**, wherein reacting the insoluble phytic acid salts in the presence of polar protic solvents comprises reacting in the presence of ethanol and water.

5. The method of claim **1**, wherein the ethanol processing fluid comprises fermentation slurry, beer, whole stillage, centrate, thin stillage, backset, or mixture thereof.

6. The method of claim **1**, wherein adding comprises introducing the phytase into the ethanol processing fluid at a temperature from about 20° C. to about 80° C.

7. The method of claim **1**, wherein adding comprises introducing the phytase for a residence time sufficient for complete reaction of the phytase with the insoluble phytic acid salts.

8. The method of claim **1**, wherein adding comprises introducing the phytase for a residence time from about 2 minutes to about 1200 minutes.

9. The method of claim **1**, further comprising analyzing an insoluble deposit in the equipment.

10. The method of claim **9**, wherein analyzing comprises detecting phytic acid or phytate.

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11. The method of claim 10, wherein analyzing comprises detecting magnesium phytate.

12. The method of claim 1, comprising adding phytase to a concentration of 500 ppm or less in the ethanol processing fluid.

13. The method of claim 1, comprising adding phytase to a concentration of 2500 U/L ppm or less in the ethanol processing fluid.

* * * * *

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Addendum 4: U.S. Patent No. 8,609,399

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ISSUE DATE: ***December 17, 2013***

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Certifying Officer

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(12) **United States Patent**
Johnson et al.

(10) **Patent No.: US 8,609,399 B2**
(45) **Date of Patent: *Dec. 17, 2013**

(54) **REDUCING INSOLUBLE DEPOSIT FORMATION IN ETHANOL PRODUCTION**

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(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

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(51) **Int. Cl.**
D06M 16/00 (2006.01)

(52) **U.S. Cl.**
USPC **435/264**; 134/18; 134/22.19

(58) **Field of Classification Search**
None
See application file for complete search history.

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(57) **ABSTRACT**

The present inventors have surprisingly discovered that phytic acid tenaciously precipitates with soluble metals in food or fuel ethanol-processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting phytic acid salts or phytates to inorganic phosphates to improve metal solubility and reduce deposition within processing equipment.

35 Claims, No Drawings

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REDUCING INSOLUBLE DEPOSIT FORMATION IN ETHANOL PRODUCTION

This application is a continuation of U.S. application Ser. No. 13/274,075, filed Oct. 14, 2011, which is a continuation of U.S. application Ser. No. 11/873,630, filed Oct. 17, 2007, now U.S. Pat. No. 8,039,244, and which applications are incorporated herein by reference. A claim of priority, to the extent appropriate, is made.

FIELD OF THE INVENTION

The present inventors have surprisingly discovered that the phytic acid tenaciously precipitates with soluble metals in food or fuel ethanol-processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting phytic acid salts or phytates to inorganic phosphates to improve metal solubility and reduce deposition within processing equipment.

BACKGROUND

Fermentation of sugars and polysaccharides into alcohol is a rapidly developing technology for producing liquid fuel, such as gasohol or E85, which are the most common examples in the United States and contain varying amounts of ethanol and gasoline. Billions of gallons of fuel ethanol are produced every year through the fermentation of grains, plants and feedstock, primarily corn. Other types of feedstock such as sugar care and cellulose are also increasing in importance.

Ethanol producers have found scale deposits on processing equipment at several stages of ethanol processing. These scale deposits are known to impede heat transfer and flow, and interfere with the proper operation of mechanical devices used in ethanol processing. The deposits tend to be most severe or tenacious on hot surfaces, and where the pH of the processing liquid is highest (about 4.5), but deposits may also form at lower pH values and on cooler surfaces. There remains a need for methods and compositions for reducing this scale formation.

SUMMARY

The present inventors have unexpectedly discovered that phytic acid tenaciously precipitates with soluble metals in food or fuel ethanol-processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting phytic acid salts or phytates to inorganic phosphates to improve metal solubility and reduce deposition within processing equipment.

In an embodiment, the present method can reduce insoluble deposit formation in equipment that contacts food or fuel ethanol-processing fluids. The method can include: adding an agent to the ethanol-processing fluids after fermentation; converting the insoluble material to a soluble residue by action of the agent; and removing the soluble residue from the equipment that contacts the ethanol-processing fluids. The method can also include identifying the insoluble deposit from the ethanol-processing fluids.

In an embodiment, the present method includes adding an enzyme with phytase activity to the ethanol-processing fluids after fermentation; converting the phytate to orthophosphate by action of the enzyme; and removing the soluble orthophosphate from the equipment that contacts the ethanol-processing fluids. This embodiment can also include identifying the insoluble deposit from the ethanol-processing fluids as phytic acid or a salt of phytic acid.

DETAILED DESCRIPTION

Definitions

As used herein, the term "mash" refers to a mixture or slurry of milled grain, process water and an enzyme such as alpha amylase, after the mixture has been subjected to a high temperature pressure "cook" and introduced into fermentation tank during ethanol processing.

"Cook water" refers to process water generated and/or used during cook process, where the starch content of milled grain is physically and chemically prepared for fermentation, typically by application of heat and by the action of enzymes such as amylase.

"Liquefaction" or "liquefy" means a process by which starch is converted to shorter chain and less viscous dextrins. Generally, this process involves gelatinization of starch simultaneously with or followed by the addition of enzymes such as amylase.

The term "liquefaction slurry" refers to combination of hot slurry, and the slurries from primary and secondary liquefaction produced during ethanol processing. Hot slurry is formed when milled grain is first mixed with process water and the formed slurry is treated with an enzyme such as α-amylase and then heated to temperatures of up to 190° F. to reduce the viscosity of the slurry. The slurry is then pumped through a pressurized jet cooker for flash condensation during primary liquefaction. After flash condensation and cooling, the primary liquefaction slurry is held at high temperature for one to two hours to provide enough time for the amylase to fully break down the starch into short chain dextrins. During secondary liquefaction or "saccharification", a second enzyme (such as glucoamylase) is added, and the formed slurry is moved into fermentation tanks. Liquefaction and saccharification may take place successively or simultaneously.

"Fermentation" refers to a process by which the sugars in the slurry or mash from liquefaction/saccharification are converted into alcohol by the action of yeast in the fermentation tanks or fermentors. The mash is allowed to ferment for 50-60 hours, resulting in a mixture that contains about 15% ethanol as well as the solids from the grain and added yeast, i.e. the "fermentation slurry." Once fermentation is complete, the mash or slurry is called "beer" and is moved into beer wells to be used for ethanol distillation and recovery.

The term "whole stillage" refers to the mash or solids remaining after ethanol is removed from beer or beer mash using a stripper column. The term is used interchangeably with the term "thick stillage." Whole stillage is typically 11% to 14% solids and contains all of the other non-starch components of the grains that pass through the process (germ, protein, gluten, hull & fiber etc.).

"Thin stillage" refers to the liquid removed from the mash in ethanol production. Thin stillage is about 5% dry matter and about 95% water. Thin stillage can be reintroduced into the cooking and distillation processes to extract additional ethanol. Thin stillage that is recycled to the beginning of the dry-grind process is known as "backset" and is used to conserve water used in processing.

The term "beerstone" refers to a hard organometallic scale deposited on fermentation equipment and that is primarily

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calcium oxalate. Although beerstone is a commonly formed deposit during ethanol processing, not all solid deposits formed are beerstone.

The term "phytase unit" refers to the amount of phytase enzyme that can liberate one micromole of ortho-phosphate from insoluble phytate in one minute, assuming optimal conditions of temperature and pH.

Methods of the Invention

The commercial processing of ethanol produces aqueous slurries of plant grains and fibers that release phytic acid. The present inventors have unexpectedly discovered that the phytic acid tenaciously precipitates with soluble metals in the processing fluid, producing insoluble organometallic salt deposit or scale on the processing equipment that must be removed in order to facilitate further ethanol processing. The present invention relates to converting insoluble phytic acid salts (i.e., phytate) to soluble inorganic phosphates and an organic compound (i.e., inositol), which can improve metal solubility and reduce deposition within processing equipment.

The present invention provides a method for reducing or even preventing the formation of insoluble material, deposits, or scale on equipment used in processing of food or fuel ethanol. According to the method, the deposit or the material forming the deposit can be converted into a soluble material by the action of an agent capable of degrading or breaking down the insoluble deposit or material that forms the deposit. The soluble material can then be easily removed from the equipment or processing system, by standard methods or conventional means known to those of skill in the art. The method can include identifying content of the deposit.

In an embodiment, the method of the present invention includes reducing the formation of insoluble deposits during ethanol processing. During commercial food and fuel ethanol processing, the aqueous slurries of plant grains and fibers produce acidic residues that interact with soluble metals in ethanol-processing fluids to produce organometallic precipitates. Many of these precipitates are insoluble solids that deposit as scale on processing equipment and interfere with downstream processing of ethanol, by impairing heat transfer and causing production interruptions. In an aspect, the solid deposit formed in this manner is beerstone, composed primarily of calcium oxalate. In another aspect, the aqueous slurries of plant grains and fibers form phosphate salts with dissolved metals present in ethanol processing fluid, such as salts of magnesium or calcium phosphate, for example. Various types of phosphate salts can be formed during ethanol processing including, without limitation, newberyite, bobierite, struvite (Mg salts), brushite, fluorapatite, hydroxyapatite (Ca salts), etc. In an aspect, the phosphate salt is a salt of a dissolved metal and phytic acid ($C_6H_{18}O_{24}P_6$; myoinositol hexakisphosphate, a phosphate ester of inositol) that is released by the plant grains and fibers present in the aqueous slurries. In another aspect, the phosphate salt is magnesium phytate. Phytate salts have been shown to form tenacious or insoluble precipitates in the presence of polar protic solvents such as water and ethanol, both of which are present in various concentrations during ethanol processing. Therefore, in an aspect, the method of the present invention provides for reducing or removing phytate that can deposit on ethanol processing equipment.

In an embodiment, the method of the present invention includes identifying the insoluble material formed during ethanol processing. Many different organometallic salts may be formed by the plant grains and fibers present in ethanol processing fluids. The solubility products of each salt may vary with processing conditions such as temperature and pH. Accordingly, the method can include chemically and/or geologically identifying the deposit or materials susceptible to deposit, which can aid in reducing or removing the insoluble deposit from the processing equipment and processing fluids. The insoluble material may be identified by standard methods known to those of skill in the art, including dry analysis methods such as x-ray fluorescence (XRF) or oxidation, followed by elemental analysis, for example, or wet analysis methods such as acid-base neutralization reactions, for example. In an aspect, the insoluble material is identified as a phosphate salt. In another aspect, the insoluble material is identified as a phytate salt, and in yet another aspect, the insoluble deposit is identified as magnesium phytate.

A considerable amount of the phosphorus or phosphate content in plant grains and fibers is in the form of phytic acid (as identified by standard analysis methods such as high temperature sample oxidation to ash), and commercial processing of these plant grains and fibers leads to release of phytic acid. These phytic acid concentrations in liquids, such as ethanol-processing fluids, can be high enough to cause precipitation of metal phytate salts, such as magnesium phytate, and subsequent deposit formation, in ethanol processing equipment. The formed phytates can impair heat transfer and cause production interruptions. A small amount of phytic acid is naturally broken down into soluble byproducts (i.e., soluble phosphates) during fermentation, but a large quantity (i.e., approximately 30-35%) of the phosphorus or phosphate in the stillage and syrup remains as phytic acid or phytate.

Metal phytate salts are generally much less soluble than the corresponding metal phosphates. For example, magnesium phytate is more than an order of magnitude less soluble than magnesium phosphate, and therefore, tends to precipitate out more readily than the more soluble magnesium phosphate. One way of causing phytate to precipitate is to heat a stable solution of magnesium phytate. Because phytate is less soluble at higher temperatures, a temperature is eventually reached where precipitation occurs, and this temperature is lower than the temperature at which magnesium phosphate would precipitate out. The temperature at which precipitation occurs is a function of pH and concentration of magnesium and phosphate or phytate ions. Assuming similar pH conditions, a solution of magnesium phosphate must be heated to about 40° C. more than a magnesium phytate solution, in order for the phosphate salt to precipitate out, even where the concentration of magnesium and phosphate ions were far greater than the concentration of magnesium and phytate ions, as shown in Table 1:

TABLE 1

Solubility of Magnesium with Phosphate and Phytate

Solution	pH	Temp (° C.)	Precipitate (+/−)
2100 ppm Mg^{2+}	5.2	40	−
8400 ppm PO_4^{3-}	5.2	60	−
(as phosphate)	5.2	90	+
	5.76	60	−
	5.76	80	++
800 ppm Mg^{2+}	4.23	80	−
3600 ppm PO_4^{3-}	4.52	60	−
(as phytate)	4.52	80	+
	4.97	40	−
	4.97	60	++
	5.36	21	−
	5.36	40	++
	5.6	21	+

Furthermore, the presence of ethanol also reduces the solubility of magnesium phytate. Table 2 indicates that precipita-

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tion of the phytate salt occurred at much lower temperature in the presence of ethanol, even at very similar pH. In the table, the water and ethanol columns indicate the temperature at which a precipitate became visible in a solution containing 800 ppm Mg^{2+} and 3600 ppm phytic acid.

TABLE 2

Solubility in Presence of Ethanol

pH	Temperature (° C.; in 100% water solution)	Temperature (° C.; in 13% ethanol/87% water solution)
4.35		40
4.52	80	
4.53		30
4.6		21
4.97	60	
5.36	40	
5.6	21	

In an embodiment, the present invention provides a method for reducing or removing insoluble material, such as phytate precipitates, by the action of an agent added to ethanol processing fluids. In an aspect, the agent is an acidic compound that can break down organic phosphates and phosphonates into soluble inorganic phosphates in the presence of a strong oxidizer or oxidizing agent. For example, a persulfate can degrade the insoluble phytate through acid digestion. In another aspect, the agent is an acidic compound that, in combination with ultraviolet light, can break down organic phosphates and phosphonates into soluble inorganic phosphates. In an embodiment, the agent is an enzyme capable of digesting or degrading (e.g., hydrolyzing) organic phosphates or phosphonates into soluble inorganic phosphates and an organic compound. For example, the agent can be a phytase, which can hydrolyze phytate to inorganic phosphate and inositol.

Phytase is an enzyme known to be capable of breaking down the phytic acid found in plant material. It is currently used primarily in animal feed applications, where it helps convert insoluble organic phosphates into soluble phosphorus that is more readily available to the animal's digestive system, and thereby also reduces environmental contamination by insoluble phosphate salts such as phytates. In ethanol processing, the phytase has been used to increase the bioavailability of phosphorus for the action of yeast in pre-saccharification and fermentation. Similarly, phytase has also been used in the liquefaction stage, or prior to fermentation to improve the activity of α-amylase.

Phytase is commercially available and can be derived from a variety of sources. In an aspect, the phytase is obtained from plants or microorganisms, such as bacteria, or from fungi, such as yeast or filamentous fungi, as disclosed in U.S. Patent Pub. No. 20050272137, for example, and incorporated herein by reference. Plant phytases may be derived from wheat-bran, maize, soybean, or lily pollen. Bacterial phytases may be derived from various bacterial sources including, without limitation, *Bacillus*, *Pseudomonas*, or *Escherichia*, preferably *B. subtilis* or *E. coli*. In another aspect, the phytase is a yeast phytase derived from *Saccharomyces* or *Schwanniomyces*, preferably *Saccharomyces cerevisiae* or *Schwanniomyces occidentalis*. In yet another aspect, phytases may be derived from filamentous fungi, including, but not limited to, species from the genus *Aspergillus*, *Thermomyces*, *Myceliophthora*, *Manascus*, *Penicillium*, *Peniophora*, *Agrocybe*, and the like.

Suitable commercially available phytases include, without limitation, those sold under the tradenames MAXALIQ™ ONE, by Genencor (Beloit, Wis.), RONOZYME® P5000 by Novozymes (Denmark), PHYTASE 5000L by DSM Food Specialties (France), NATUPHOS® 5000 by BASF (Germany), and PHYZYME™ XP 10000 by Danisco Animal Nutrition (St. Louis, Mo.). Suitable commercially available phytase enzyme may also be obtained from suppliers including, without limitation, Deerland Enzymes (Kennesaw, Ga.).

In an embodiment, phytase is added to ethanol processing equipment and/or processing fluid at a time point and under conditions required for the particular type of equipment or stage of ethanol processing. In an aspect, phytase is added to fermentation fluid to reduce formation of insoluble deposits in downstream ethanol processing equipment. In another aspect, phytase is added to ethanol processing fluids downstream of the fermentation process, such as, for example, beer, whole stillage, thin stillage, backset, centrate, or a mixture of these fluids. In an embodiment, phytase is added to thin stillage or backset. In an aspect, phytase is added to thin stillage or backset in line, i.e. the enzyme is introduced directly into thin stillage or backset-containing equipment during ethanol processing. In another aspect, phytase is added to thin stillage or backset offline, i.e. the enzyme is added to thin stillage or backset contained in a separate vessel or tank. Phytase-treated thin stillage or backset can then be cycled back into processing lines from the vessel or tank.

In an embodiment, the present invention provides a method in which the agent or enzyme is introduced into the ethanol-processing fluid under optimal conditions of temperature and pressure. Where the agent is phytase, the term "optimal conditions" refers to those conditions of concentration, temperature, residence time or reaction time, and pH that allow sufficient reaction with soluble phytate, phytate suspension, phytate precipitate, or insoluble phytate scale that reduces the level of the phytase deposit to an amount acceptable for operation of the ethanol plant or process. In an embodiment, the conditions provide for complete hydrolysis of soluble phytate and phytate suspension.

In an aspect, the phytase is added to the ethanol processing fluid at temperatures of about 20° C. to about 80° C., for example, about 20° C. to about 77° C., about 40° C. to about 65° C., or about 30° C. to about 55° C. (e.g., 52° C.). In an aspect, the phytase is added to the ethanol processing fluid at temperatures sufficient to allow the reaction between phytate and phytase to proceed to completion without degrading the enzyme. In another aspect, the phytase is added to the ethanol processing fluid at pH of about 3 to about 9, for example, about 4.0 to about 5.0, about 4.0 to about 5.5, or about 4.0 to about 5.3. In yet another aspect, the phytase is added at a pH of 4.0, and the reaction is conducted at temperatures of about 40° C. to about 65° C., about 20° C. to about 77° C., or about 30° C. to about 55° C. (e.g., 52° C.).

In an aspect, the phytase is added to the ethanol processing fluids at a concentration of about 100 ppm to about 500 ppm. In another aspect, the phytase is added at a concentration of 100 ppm, and in yet another aspect, the phytase is added at a concentration of 500 ppm. The phytase can be added at concentrations expressed in phytase units. A unit of activity (U) is the amount of phytase that can release 1 μmol of orthophosphate per minute from excess phytic acid/phytate, at a temperature of 37° C. and a pH of about 5.5. Therefore, in an aspect, the phytase is added at a concentration of about 500 U/L to about 2500 U/L. In another aspect, the phytase is added at a concentration of 100 U/L, and in yet another aspect, the phytase is added at a concentration of 2500 U/L.

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In an embodiment, the phytase is added to ethanol processing fluids at lower concentrations, and the reaction is allowed to proceed over longer periods of time. Extending the reaction time or residence time allows smaller amounts of enzymes to be used, making ethanol processing more economical. In an aspect, the phytase is added to the ethanol processing fluids for a residence time sufficient for complete reaction of the phytase with the insoluble phytate. In another aspect, the phytase is added for a residence time of about 2 minutes to about 1200 minutes, for example, about 3 minutes to about 200 minutes, or about 3 minutes to about 40 minutes.

As shown in Table 3, low concentrations of phytase can release significant amounts of ortho-phosphate, if reacted over longer periods of time. For example, 5 ppm of phytase will release approximately 900 ppm of ortho-phosphate when reacted over a 20 hour time period. In contrast, 100 ppm of phytase releases 400-500 ppm of ortho-phosphate in just 10 minutes.

TABLE 3

Orthophosphate Formation at Low Doses of Phytase

Time	Amount of Released Ortho-Phosphate (ppm)		
(min)	5 ppm phytase	10 ppm phytase	20 ppm phytase
20	73	92	109
60	143	232	387
140	260	436	732
260	377	636	933
1200	894	1460	1713

Therefore, in an aspect, the phytase is added at a concentration of about 5 ppm to about 20 ppm. In another aspect, the phytase is added at a concentration of 5 ppm. In a further aspect, the phytase is added at a concentration of 10 ppm, and in a yet further aspect, the phytase is added at a concentration of 20 ppm. These concentrations may also be expressed in phytase units such that, in an aspect, the phytase is added at a concentration of about 25 U/L to about 100 U/L. In another aspect, the phytase is added at a concentration of 25 U/L. In a further aspect, the phytase is added at a concentration of 50 U/L and in a yet further aspect, the phytase is added at a concentration of 100 U/L.

In an embodiment, the present invention provides a method in which the agent added to the ethanol processing fluid converts soluble phytate, phytate suspension, phytate precipitate, or phytate scale into soluble orthophosphate. In an aspect, the agent is an enzyme such as phytase, which specifically hydrolyzes phytate. The enzyme can be used to convert phytate materials into soluble phosphates that are easily removed from the processing fluid, if necessary. Assuming identical conditions of pH (5.3) and identical concentrations of Mg^{2-} ions (1048 ppm) and phytic acid (4702 ppm), samples of processing fluid treated with phytase remained clear (i.e., phosphates remain in solution without precipitating out). However, samples of processing fluid not exposed to phytase showed progressively more precipitation of magnesium phytate as the temperature is increased, as shown in Table 3 below.

TABLE 4

Enzyme Treatment Prevents Precipitation

Enzyme Treated? (Y/N)	Temp (° C.)	Precipitate? (+/−)
Y	20	−
Y	40	−
Y	80	−
Y	100	−
N	20	+
N	40	++
N	60	+
N	80	+++

The following examples are provided to illustrate various aspects of the invention, and should not be construed to limit the invention. A person of skill in the art will recognize that various modifications may be made to the examples without departing from the scope of the present invention.

EXAMPLES

Example 1

Conventional Analyses Indicate that Deposits in the Beer Column Contain Phosphorus, but do not Reveal that the Phosphorus is in Phytate

The content of various solid deposits formed during ethanol fermentation can be determined using standard methods. It was previously thought that the solid deposits in the beer column were primarily beerstone (i.e. calcium oxalate). The unexpected results shown in the following examples demonstrate, however, that a large percentage of the solid deposits found in the beer column are in the form of phosphates, i.e. P_2O_5.

Materials and Methods

To determine the content of the solid deposit in the beer column, X-ray fluorescence (XRF) analysis was used. A sample of solid deposit from the beer column was collected and air-dried. A portion of the sample was ground to approximately 400 mesh using a steel swing mill, and the ground sample was analyzed by XRF. Using standard XRF procedures, it was possible to determine the presence of 31 major, minor and trace elements to a relative precision/accuracy of approximately 5-10% for major and minor elements and approximately 10-15% for trace elements. A replicate sample was analyzed, along with a standard reference material ("SY3", a CANMET standard rock or geological sample) to demonstrate analytical reproducibility as well as analytical accuracy for a geological standard.

Results

The content of the solid deposit in the beer column was as shown in Table 5. Major elements in the solid deposit include magnesium (as MgO) and phosphorus (as P_2O_5), while potassium and calcium (as their respective oxides K_2O and CaO) occur as minor elements. Trace amounts of zinc were also detected. Major and minor elements were represented as weight percentages of the deposit, while trace elements were represented in ppm units.

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TABLE 5

Deposit Analysis	
Element	Content
Magnesium (as MgO)	12.5 wt %
Phosphorus (as P_2O_5)	35.7 wt %
Potassium (as K_2O)	2.54 wt %
Calcium (as CaO)	2.76
Zinc	18447 ppm

Conclusions

The results in Table 5 demonstrate that the majority of the solid deposits are present as phosphate (rather than as the expected beerstone or calcium oxalate). XRF analysis of solid deposits shows a large concentration of magnesium and phosphorus present in the oxide form, but does not distinguish between different chemical forms of phosphorus or phosphate salts, and does not specify if some of the magnesium was actually present as a phosphate salt (i.e. magnesium phytate). Because different forms of phosphorus and phosphate salts have different solubilities and because the phytase has different activities on different forms of phosphate, it is useful to distinguish between the various forms.

Example 2

The Deposits Include Substantial Amounts Phosphorus in Phytate

Because the activity of the phytase on the solid deposits is dependent on the type of phosphorus or phosphate salts present, wet analysis of various deposit samples was used to determine the different forms of phosphorus and phosphate salts present in the solid deposits of the beer column.

Materials and Methods

Deposit samples from ethanol plants were dissolved in a weighed amount of acid, the acid was neutralized and the solution was diluted to a known volume. A portion of the neutralized sample was then subjected to a standard test for ortho-phosphate (PO_4^{3}), i.e. the Hach test. Total phosphorus was determined by acid oxidation with persulfate, followed by the reactive phosphorus test. Organically bound ortho phosphate was then determined by subtracting the acid-hydrolysable phosphorus content, and was reported as PO_4^{3-} or ortho-phosphate.

A separate portion of the neutralized solution was treated with phytase (at a concentration of 500 ppm and 41° C. for 20-30 minutes). Under these conditions, phytate present in the solution was converted to ortho-phosphate. The amount of ortho-phosphate (PO_4^{3-}) present after phytase treatment represents the total phosphorus content of the deposit (total P as PO_4^{3-}), i.e. original ortho phosphate and phytate phosphorus.

Results

As indicated in Table 6, deposit samples from an evaporator at one plant, and samples from cook water lines and liquefaction pumps at a second plant had between 60% and 75% of their phosphorus content as phytate, confirming that the deposit in the beer column included primarily magnesium phytate.

TABLE 6

Comparison of Deposit Analysis by XRF and Wet Methods

	Content by XRF		Content by Wet Analysis	
Sample	% MgO	% P_2O_5	% PO_4^{3-}	Total % P as PO_4^{3-}
Plant 1 Evaporator (1)	14.6	36.8	7.4	37.4
Plant 1 Evaporator (2)	11.3	23.0	1.9	27.7
Plant 1 Evaporator (3)	10.5	25.5	5.9	30.9
Plant 2 Cook Water	14.0	35.5	2.4	36.0
Plant 2 Pump	16.7	35.2	9.1	38.0

Conclusions

The results in Table 6 demonstrate that the solid deposits were present as phytate salt, i.e. magnesium phytate. This is an important discovery because the activity of the phytase added to various structures in an ethanol plant depends on the presence of phosphate primarily in the phytate form. Solid deposits that exist primarily as phytate can be dissolved by the action of the phytase.

Example 3

Phytase Reduces the Phytate Concentration in Thin Stillae, which is the Source of the Deposits

Materials and Methods

The amount of phosphorus (present as ortho phosphate) in the thin stillage fraction collected from an ethanol plant will decrease once phytase has been added. To determine the decrease in ortho-phosphate concentration over time as a result of enzyme activity, thin stillage fractions were collected and treated with phytase at temperatures from 43° C. to 62° C., at a pH of 4.0. The phytase concentration was either 100 ppm or 500 ppm. After adding the enzyme, ortho-phosphate concentration was measured at various time points (e.g., from 3 minutes to 20 minutes). Any phosphorus present as phytate (about 40-50% of the total phosphate) was converted to ortho phosphate (PO_4^{3-}) by the action of the phytase, with more phytate converted over increasing periods of time. The concentration of ortho-phosphate in the thin stillage was therefore a measure of enzyme activity.

Results

As demonstrated in Table 7, at an enzyme concentration of 500 ppm and a temperature of 43° C., the reaction was essentially complete in 5 minutes, indicating that phytate was completely converted to ortho phosphate. At an enzyme concentration of 100 ppm, and a temperature of 62° C., the reaction continued for 20 minutes.

TABLE 7

Conversion of Phytate in Thin Stillage to Ortho Phosphate

Sample/Run (min)	Enzyme Conc. (ppm)	Temp (° C.)	Phosphate Conc. (ppm)	Released PO_4^{3-} Conc. (ppm)
Distilled Water (zero)	—	—	0	—
Std. 1 - 640 ppm PO_4^{3-}	—	—	732	—
Std. 2 - 1280 ppm PO_4^{3-}	—	—	1234	—

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TABLE 7-continued

Conversion of Phytate in Thin Stillage to Ortho Phosphate				
Sample/Run (min)	Enzyme Conc. (ppm)	Temp (° C.)	Phosphate Conc. (ppm)	Released PO_4^{3-} Conc. (ppm)
Run 1				
5 min	500	45.5	3313	1463
10 min	500	43.3	3385	1536
Std 2			1242	—
Run 2				
3 min	100	63.3	1688	137
5 min	100	62.8	2146	595
10 min	100	62.2	2214	663
20 min	100	61.0	2859	1309

Conclusions

The results shown in Table 7 indicate that substantially all of the phosphorus present in the phytate form was converted and released as ortho-phosphate by the action of the phytase. About half of the total phosphorus content of the thin stillage was in the form of phytate, and complete reaction of the enzyme with the phosphorus resulted in the conversion of phytate and release of ortho-phosphate. The amount or concentration of released ortho-phosphate therefore provides a measure of the activity of the phytase.

Example 4

Increased Solubility on Conversion of Phytate to Ortho-Phosphate

The solid deposits formed in various structures in an ethanol plant include primarily insoluble magnesium phosphate salts, including magnesium phytate. The following example illustrates that the solubility of these salts is unexpectedly increased in the presence of phytase.

Materials and Methods

To determine the effect of phytase on increasing the solubility of magnesium phytate, two solutions of magnesium phytate were prepared by mixing magnesium salt with phytic acid in two separate test tubes. The solution in one test tube was treated with phytase, while the other was left untreated. The two solutions were maintained at the same temperature and pH and had identical magnesium and total phosphorus content.

Results

The action of the phytase on magnesium phytate converted the insoluble phytate salt into soluble ortho phosphate, which can then be readily removed from pumps, lines and evaporators in an ethanol plant. FIG. 1 illustrates the differences in solubility of phytic acid solutions with and without the action of phytase.

The enzyme-treated solution in one test tube remained clear, indicating that all the phytate was converted to soluble ortho phosphate. The untreated solution showed precipitate formation, indicating the presence of insoluble magnesium phytate. This demonstrated that the enzyme completely converted the phytate to ortho-phosphate and greatly increased the solubility of the magnesium salt.

Conclusion

Phytase can prevent solid deposits of magnesium phosphate salts from forming in various structures in an ethanol plant.

Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art. Although any methods, devices and material similar or equivalent to those described herein can be used in practice or testing, the methods, devices and materials are now described.

All publications and patent applications in this specification are indicative of the level of ordinary skill in the art to which this invention pertains and are incorporated herein by reference in their entireties.

In this specification and the appended claims, the singular forms "a," "an," and "the" include plural reference, unless the context clearly dictates otherwise. Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood to one of ordinary skill in the art.

The various embodiments described above are provided by way of illustration only and should not be construed to limit the invention. Those skilled in the art will readily recognize various modifications and changes that may be made to the present invention without following the example embodiments and applications illustrated and described herein, and without departing from the true spirit and scope of the present invention without following the example embodiments and applications illustrated and described herein, and without departing from the true spirit and scope of the present invention, which is set forth in the following claims.

The claimed invention is:

1. A method of reducing formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during the production of a quantity of ethanol, wherein the production of a quantity of ethanol comprises a fermentation step and wherein the fuel ethanol processing plant comprises a piece of heat transfer equipment, the method comprising:

providing an additive in an ethanol processing fluid in the plant, wherein the ethanol processing fluid comprises an amount of phytic acid and/or salts of phytic acid, and wherein the additive comprises phytase,

wherein the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid by breaking down the phytic acid and/or phytic acid salts,

wherein providing the additive comprising phytase in the ethanol processing fluid causes a reduction of the formation of insoluble deposits of phytic acid and/or salts of phytic acid in a piece of heat transfer equipment in the plant, and

wherein the reduction in the formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during production of the quantity of ethanol is accomplished substantially without the addition of an acidic compound that can break down organic phosphates and phosphonates into soluble inorganic phosphates in the presence of an oxidizer, oxidizing agent, or ultraviolet light.

2. A method of reducing formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during the production of a quantity of ethanol, wherein the production of a quantity of ethanol comprises a fermentation step and wherein the fuel ethanol processing plant comprises a beer column and a piece of heat transfer equipment, the method comprising:

providing an additive in an ethanol processing fluid in the plant, wherein the ethanol processing fluid comprises an

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amount of phytic acid and/or salts of phytic acid, and wherein the additive comprises phytase,

wherein the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid by breaking down the phytic acid and/or phytic acid salts,

wherein providing the additive comprising phytase in the ethanol processing fluid causes a reduction of the formation of insoluble deposits of phytic acid and/or salts of phytic acid in a piece of heat transfer equipment in the plant, and

wherein the pH of the ethanol processing fluid in the beer column is 4.5 or higher during production of the quantity of ethanol.

3. The method of claim 1, wherein the additive comprising phytase is added to an ethanol processing fluid prior to fermentation.

4. The method of claim 1, wherein the additive comprising phytase is added to an ethanol processing fluid following fermentation.

5. The method of claim 1, wherein the plant further comprises a beer column and wherein providing the ethanol processing fluid comprising phytase also causes the amount of insoluble phytic acid and/or salts of phytic acid deposited in the beer column to be reduced.

6. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 500 ppm or less.

7. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 100 ppm or less.

8. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 20 ppm or less.

9. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 10 ppm or less.

10. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 2500 U/L or less.

11. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 500 U/L or less.

12. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 100 U/L or less.

13. The method of claim 5, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 50 U/L or less.

14. The method of claim 2, wherein the additive comprising phytase is added to an ethanol processing fluid prior to fermentation.

15. The method of claim 2, wherein the additive comprising phytase is added to an ethanol processing fluid following fermentation.

16. The method of claim 2, wherein providing the ethanol processing fluid comprising phytase also causes the amount of insoluble phytic acid and/or salts of phytic acid deposited in the beer column to be reduced.

17. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 500 ppm or less.

18. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 100 ppm or less.

19. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 20 ppm or less.

20. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 10 ppm or less.

21. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 2500 U/L or less.

22. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 500 U/L or less.

23. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 20 ppm or less.

24. The method of claim 16, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount 50 U/L or less.

25. A method of reducing formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during the production of a quantity of ethanol, wherein the production of a quantity of ethanol comprises a fermentation step and wherein the fuel ethanol processing plant comprises a beer column and a piece of heat transfer equipment, the method comprising:

providing an additive in an ethanol processing fluid in the plant, wherein the fluid comprises an amount of phytic acid and/or salts of phytic acid, and wherein the additive comprises phytase,

wherein the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid by breaking down the phytic acid and/or phytic acid salts,

wherein providing the additive comprising phytase in the ethanol processing fluid causes a reduction of the formation of insoluble deposits of phytic acid and/or salts of phytic acid in a piece of heat transfer equipment in the plant, and

wherein providing the ethanol processing fluid comprising phytase also causes the amount of insoluble phytic acid and/or salts of phytic acid deposited in the beer column to be reduced,

wherein the reduction in the amount of insoluble phytic acid and/or salts of phytic acid formed in the heat transfer equipment and deposited in the beer column is accomplished substantially without the addition of an acidic compound that can break down organic phosphates and phosphonates into soluble inorganic phosphates in the presence of an oxidizer, oxidizing agent, or ultraviolet light,

wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing

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fluid, the phytase is present in the ethanol processing fluid in an amount of 500 ppm or less, and

wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 2500 U/L or less.

26. The method of claim **25**, wherein the additive comprising phytase is added to an ethanol processing fluid prior to fermentation.

27. The method of claim **25**, wherein the additive comprising phytase is added to an ethanol processing fluid following fermentation.

28. The method of claim **25**, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 100 ppm or less.

29. The method of claim **25**, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 20 ppm or less.

30. The method of claim **25**, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 10 ppm or less.

31. The method of claim **25**, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 500 U/L or less.

32. The method of claim **25**, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 100 U/L or less.

33. The method of claim **25**, wherein when the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid, the phytase is present in the ethanol processing fluid in an amount of 50 U/L or less.

34. A method of reducing formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during the production of a quantity of ethanol, wherein the production of a quantity of ethanol comprises a fermentation step and wherein the fuel ethanol processing plant comprises a piece of heat transfer equipment, the method comprising:

providing an agent in an ethanol processing fluid in the plant, wherein the ethanol processing fluid comprises an amount of phytic acid and/or salts of phytic acid, and wherein the agent comprises phytase,

wherein the phytase reduces the amount of phytic acid and/or phytic acid salts in the ethanol processing fluid by breaking down the phytic acid and/or phytic acid salts into soluble inorganic phosphates and an organic compound,

wherein providing the agent comprising phytase in the ethanol processing fluid causes a reduction of the formation of insoluble deposits of phytic acid and/or salts of phytic acid in a piece of heat transfer equipment in the plant, and

wherein the reduction in the formation of insoluble deposits of phytic acid and/or salts of phytic acid in fuel ethanol processing plant equipment or a portion thereof during production of the quantity of ethanol is accomplished substantially without the addition of an agent including an acidic compound that can break down organic phosphates and phosphonates into soluble inorganic phosphates in the presence of an oxidizer, oxidizing agent, or ultraviolet light.

35. The method of claim **25**, wherein the pH of the ethanol processing fluid in the beer column is 4.5 or higher during production of the quantity of ethanol.

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